Blue Thermomechanical Treatment Optimizes Fatigue Resistance and Flexibility of the Reciproc Files

Gustavo De-Deus, DDS, MSc, PhD,* Emmanuel João Nogueira Leal Silva, DDS, MSc, PhD, † Victor Talarico Leal Vieira, DDS, MSc, PhD, † Felipe Gonçalves Belladonna, DDS, MSc,* Carlos Nelson Elias, PhD, † Gianluca Plotino, DDS, MSc, PhD, $^{\parallel}$ and Nicola Maria Grande, DDS, PhD $^{\parallel}$

Abstract

Introduction: The aim of the present study was to evaluate the influence of Blue thermal treatment on the bending resistance and cyclic fatigue of conventional M-Wire Reciproc files (VDW, Munich, Germany). The roughness pattern and the microhardness of the files were also assessed. Methods: Flexibility of standard Reciproc R25 files and the corresponding Blue prototypes was determined by 45° bending tests according to the ISO 3630-1 specification. Instruments were also subjected to cyclic fatigue resistance, measuring the time to fracture in an artificial stainless steel canal with a 60° angle and a 5-mm radius of curvature. The fracture surface of all fragments was examined with a scanning electron microscope. The roughness of the working parts was quantified by using a profilometer, and the microhardness test was performed using the Vickers hardness tester. Results were statistically analyzed using the Student t test with a level of significance set at P < .05. Results: Reciproc Blue instruments presented a significantly longer cyclic fatigue life and significantly lower bending resistance than the original Reciproc instrument (P < .05). Regarding the roughness pattern, there was no significant difference between Reciproc Blue and the original Reciproc instruments (P > .05), whereas Reciproc Blue revealed significantly lower microhardness than the original Reciproc instrument (P < .05). Conclusions: Reciproc Blue nickel-titanium showed improved all-around performance when compared with conventional M-Wire superelastic nickel-titanium, demonstrating improved flexibility and fatigue resistance, and reduced microhardness while maintaining similar characteristics of the surface. (J Endod 2016; \blacksquare :1-5)

Kev Words

Blue treatment, nickel-titanium, Reciproc

Nickel-titanium (NiTi) rotary instruments have been widely used for root canal preparation. However, despite their numerous advantages,

Significance

The overall improved mechanical features of the Reciproc Blue file may impact on its clinical performance.

these instruments present risk of fracture during its use in curved canals, which might compromise the prognosis of root canal treatment (1). Different alloys and crosssectional designs have been proposed to increase the flexibility and resistance to fatigue fracture of endodontic instruments (2, 3). Thermal treatment of NiTi alloys has been successfully used to improve the mechanical properties of endodontic instruments such as fatigue resistance, flexibility, cutting efficiency, and canal centering ability (2–5). Manufacturers have introduced several thermally treated NiTi alloys (eg, controlled memory wire [CM Wire; DS Dental, Johnson City, TN], M-Wire [Dentsply Tulsa Dental Specialties, Tulsa, OK], and R-phase wire [SybronEndo, Orange, CA]) to optimize the microstructure and transformation behavior of NiTi alloys, which in turn has greater influence on the mechanical properties of NiTi files (2-5). A new generation of instruments (Vortex Blue and ProTaper Gold rotary files; Dentsply Tulsa Dental Specialties) undergo a complex heating-cooling proprietary treatment that results in a visible titanium oxide layer in the surface of the instrument. This treatment controls the transition temperatures, creating a shape memory alloy, which is claimed by the manufacturer to result in superior mechanical properties and performance of the NiTi instruments (6-8).

In addition to the thermal treatment modifications, a new kinematic (reciprocating motion) has already been shown to extend the life span of a NiTi instrument and its resistance to fatigue in comparison with continuous rotation movement (9–11). Driven by reciprocation movement, the instruments travel a shorter angular distance than rotary instruments, being subject to lower stress values and resulting in an extended fatigue life (9–11). Reciproc (VDW, Munich, Germany) and WaveOne (Dentsply Maillefer, Baillagues, Switzerland) are the main examples of commercially available systems for root canal preparation using reciprocating motion.

From the *Department of Endodontics, Fluminense Federal University, Niterói, Rio de Janeiro, Brazil; [†]Department of Endodontics, Grande Rio University, Duque de Caxias, Rio de Janeiro, Brazil; [‡]Department of Biomaterials, Military Institute of Engineering, Duque de Caxias, Rio de Janeiro, Brazil; [§]Grande Plotino & Torsello - Studio di Odontoiatria, Rome, Italy; and ^{||}Department of Endodontics, Catholic University of Sacred Heart, Rome, Italy.

Address requests for reprints to Prof Gustavo De-Deus, Av Henrique Dodsworth, 85, Apto 808, Lagoa, Rio de Janeiro, RJ 22061-030, Brazil. E-mail address: endogus@gmail.com

0099-2399/\$ - see front matter

Copyright © 2016 American Association of Endodontists. http://dx.doi.org/10.1016/j.joen.2016.10.039

Basic Research—Technology

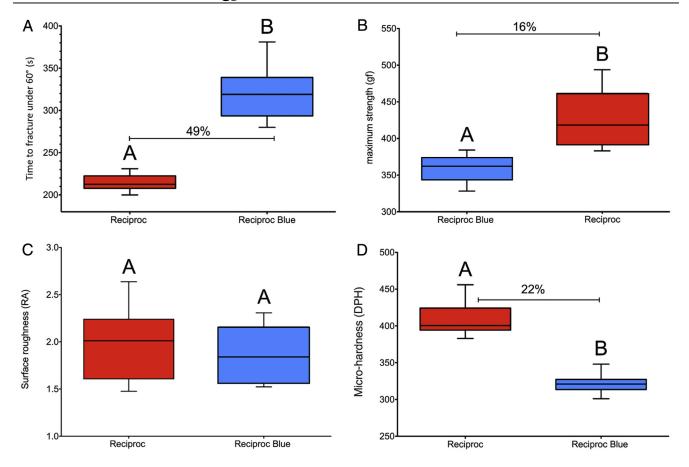


Figure 1. A box plot showing the average, median, minimal, and maximal values and the standard deviation of (A) bending resistance, (B) cyclic fatigue, (C) surface roughness, and (D) microhardness. Different letters indicate significant statistical differences between the groups (P < .05).

In the present study, the influence of Blue thermal treatment on the bending resistance and the cyclic fatigue of conventional M-Wire Reciproc files was evaluated. In addition, the roughness pattern and the microhardness of the files were assessed. The null hypotheses tested were as follows:

- There are no differences in the bending resistance between the instruments
- There are no differences in the cyclic fatigue fracture resistance between the instruments
- 3. There are no differences in the roughness pattern of the instruments
- There are no differences in the microhardness between the instruments

Materials and Methods

A sample of 30 NiTi instruments (25 mm in length) for use under reciprocation movement (Reciproc R25) and 30 prototypes of Reciproc instruments manufactured in Blue alloy (Reciproc Blue R25) were tested. All Reciproc instruments R25 had a nominal size of 0.25 mm at the tip and a taper of 0.08 mm/mm (in the apical 3 mm of the tip). For standardization and reliability of the experiment, the tested instruments were previously examined for defects or deformities under a stereomicroscope.

Bending Resistance Test

The bending resistance test was performed in 10 randomly selected instruments of each system by using a universal testing machine (DL 200 MF; Emic, São José dos Pinhais, Brazil) according to the ISO

3630-1 specification (12). A 20-N load was applied at 15 mm/min by means of a flexible stainless steel wire with one end fastened to the testing machine head and the other end attached 3 mm from the instrument tip as previously described (13). This test was conducted until the tip of each specimen underwent an elastic displacement of 45° . The force values were acquired in the 45° position, and the maximum load to bend each file was recorded.

Cyclic Fatigue Test

The cyclic fatigue test was performed by using a custom-made device like the one previously described (10). The artificial canal was manufactured with a tapered shape corresponding to the dimensions of the instruments tested. It provided a suitable simulated root canal with a 60° angle of curvature and 5-mm radius of curvature. The center of the curvature was 5 mm from the tip of the instrument, and the curved segment of the canal was 5 mm in length. The artificial canal was open in its upper part and covered with tempered glass to prevent the instruments from slipping out.

Ten instruments of each 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 presetting programs for the Reciproc system ("RE-CIPROC ALL") as recommended by the manufacturer. All instruments were driven following the manufacturer's instructions until a fracture occurred. The instruments reciprocated freely within the stainless tube (simulated canal), which was filled with glycerin to reduce friction and heat production. Each instrument was positioned in a contra-angle handpiece and introduced into the canal

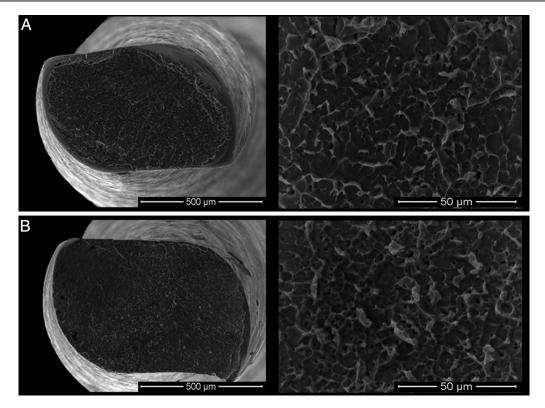


Figure 2. Scanning electron microscopic analysis of the fractured surfaces of the tested instruments after cyclic fatigue test. The images show magnifications of $(left) 200 \times$ and $(right) 1500 \times$. (A) Reciproc instrument and (B) Reciproc Blue instrument.

until the tip touched a shield positioned at the other extremity. This shield was subsequently removed once it was only used to standardize the instrument penetration into the canal. The time was recorded and stopped as soon as a fracture was detected visually and/or audibly. To limit possible human errors, video recording was performed simultaneously, and the recordings were then observed to cross-check the time of file fracture (13).

A scanning electron microscope (FEG Quanta 250; FEI, Eindhoven, Netherlands) was used to analyze the fracture surfaces and the helical shaft of the fractured instruments in order to determine the fracture mode and the occurrence of plastic deformation in the helical shaft. Different magnifications were used ($\times 200$ and $\times 1500$), and the photomicrographs were used for further analyses.

Surface Finish

The roughness of the working parts of the conventional and Blue files was quantified by using a New View 7100 Profilometer (Zygo Co, Middlefield, CT) in 5 instruments of each type. The New View is an interferometric noncontact 3-dimensional surface measurement system. The profiler gives ultraprecise 3-dimensional analyses of any surface and rapidly measures heights from 0.1 nm to 1.0 mm, with vertical resolution as low as 0.1 nm.

Roughness was quantified at the apical, middle, and coronal thirds of the instruments; 3 measurements were performed for each third in randomly selected areas for a total of 9 measurements per instrument. The groove depth value for each instrument was established as the mean of the 9 measurements.

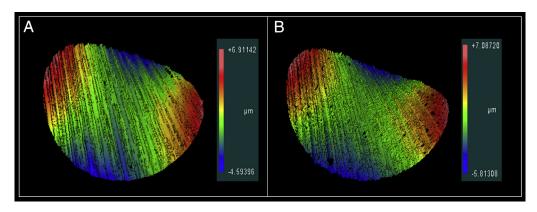


Figure 3. Instrument surface morphology obtained by interferometry. (A) Reciproc instrument and (B) Reciproc Blue instrument.

Basic Research—Technology

Vickers Microhardness Test

The microhardness test was performed using a Vickers hardness tester (Micromet 2003; Buehler, Lake Bluff, IL). Five instruments of each type were embedded in resin and then prepared for microhardness evaluation. The diamond penetrator was used with 100-g force for 15 seconds and evaluated in at magnification x 40. Ten indentations were performed on each instrument.

Statistical Analysis

Because the preliminary analysis of the raw pooled and isolated data revealed a bell-shaped distribution using the D'Agostino-Person omnibus normality test, statistical analysis was performed using a parametric method Student *t* test. The alpha-type error was set at 0.05. SPSS 11.0 (SPSS Inc, Chicago, IL) and Origin 6.0 (Microcal Software, Inc, Northampton, MA) were used as analytical tools.

Results

The average, the minimal and maximal values, and the standard deviation of the results obtained in the bending, cyclic fatigue, surface roughness, and microhardness tests are shown in the graphs of Figure 1A–D. Reciproc Blue revealed significantly lower bending resistance and longer cyclic fatigue life than the original Reciproc (P < .05). Scanning electron microscopic visual inspection of the fractured surface indicated that all instruments showed fractographic characteristics of ductile fracture. Wide-ranging forms of dimples were identified overall, and no plastic deformations in the helical shaft of the fractured instruments were observed (Fig. 2A and B).

Regarding the roughness pattern, there was no significant difference between Reciproc Blue and the original M-Wire Reciproc (Fig. 3A and B), whereas Reciproc Blue revealed significantly lower microhardness than the original Reciproc (P < .05).

Discussion

In the present study, the instruments were tested for flexibility following the American National Standards Institute/American Dental Association specification no. 28 (2002) and ISO 3630/1 (2008), which are respectively designed to measure strength under torsion and flexibility of stainless steel hand files and for instruments having a 0.02 ISO taper (12, 14).

Despite the fact that cyclic fatigue resistance tests still do not have a specification or an international standard to evaluate NiTi endodontic mechanical instruments, this test was performed in the present article using a methodology introduced by the authors and validated in many studies published in peer-reviewed journals (6, 10, 11, 13, 15-18).

The profilometer used in the present study for roughness analysis of the surfaces of the instruments is an interferometric noncontact 3-dimensional surface measurement system that gives ultraprecise 3-dimensional analyses of any surface and rapidly measures heights from 0.1 nm to 1.0 mm, with vertical resolution as low as 0.1 nm. The microhardness test was performed using a Vickers hardness tester following the methodology of previously published investigations on instruments made by NiTi alloys (19, 20).

The influence of file design on the tested parameters was virtually eliminated in the present study by testing instruments that differ only in their manufacturing process (ie, Reciproc and Reciproc Blue prototypes). The overall results of the present study showed that Reciproc instruments manufactured using the new Blue thermomechanical treatment outperformed the conventional Reciproc instruments made by M-Wire NiTi in all tests; they had similar patterns in the roughness analysis.

The results from the bending resistance test conducted in the present study showed that Blue files had a significant improvement in flexibility over the other tested commercially available Reciproc instruments. These results confirm those obtained in a previous study investigating another instrument produced both in M-Wire and Blue alloys (ie, Vortex rotary files) (21). The results were statistically significant; thus, the null hypothesis tested in the present study may be rejected.

The results of the cyclic fatigue test are strictly related with the flexibility of the instruments tested and confirmed previous studies in which NiTi Blue alloy showed enhanced mechanical properties when compared with M-Wire NiTi (6, 21). In fact, Reciproc Blue showed a significant increase in the mean time to fracture when compared with the same size traditional Reciproc files. The results were statistically significant in terms of cyclic fatigue resistance; thus, the second null hypothesis tested in the present study may be rejected.

The profilometer roughness analysis performed in the present study revealed no statistical differences between conventional Reciproc and Reciproc Blue instruments in all thirds. These results showed a high quality of manufacturing on both type of instruments and that the different thermal treatments have no influence on the surface characteristics of the instruments. The third null hypothesis tested in the present study cannot be rejected due to this specific feature.

A statistically significant difference was noted when investigating the microhardness of the 2 different instruments. The Reciproc Blue files showed less Vickers surface hardness than the traditional M-Wire files, which may indicate that this proprietary thermomechanical treatment produces a NiTi alloy that is softer and more ductile than the traditional one. These data may be also related to the fact that this softer metal has an increased flexibility and fatigue resistance, as shown herein. Also, for this feature, the fourth null hypothesis tested in the present study may be rejected.

In conclusion, Blue NiTi, a newly developed alloy that is obtained through a proprietary-specific oxide surface layer thermomechanical manufacturing process, showed overall improved performances when compared with conventional M-Wire superelastic NiTi, demonstrating improved flexibility and fatigue resistance, and reduced microhardness while maintaining similar characteristics of the surface. The findings of the current study showed that Reciproc files may benefit from this manufacturing process, but this must be confirmed by further *in vitro* and *in vivo* investigations.

Acknowledgments

The authors deny any conflicts of interest related to this study.

References

- Ankrum MT, Hartwell GR, Truitt JE. K3 Endo, ProTaper, and ProFile systems: breakage and distortion in severely curved roots of molars. J Endod 2004;30: 234-7.
- Pérez-Higueras JJ, Arias A, de la Macorra JC. Cyclic fatigue resistance of K3, K3XF, and twisted file nickel-titanium files under continuous rotation or reciprocating motion. J Endod 2013;39:1585–8.
- Lopes HP, Gambarra-Soares T, Elias CN, et al. Comparison of the mechanical properties of rotary instruments made of conventional nickel-titanium, M-wire, or nickel-titanium alloy in R-phase. J Endod 2013;39:516–20.
- Shen Y, Qian W, Abtin H, et al. Effect of environment on fatigue failure of controlled memory wire nickel-titanium rotary instruments. J Endod 2012;38:376–80.
- Pereira ES, Gomes RO, Leroy AM, et al. Mechanical behavior of M-wire and conventional NiTi wire used to manufacture rotary endodontic instruments. Dent Mater 2013;29:e318–24.
- Plotino G, Grande NM, Cotti E, et al. Blue treatment enhances cyclic fatigue resistance of vortex nickel-titanium rotary files. J Endod 2014;40:1451–3.

ARTICLE IN PRESS

Basic Research—Technology

- Hieawy A, Haapasalo M, Zhou H, et al. Phase transformation behavior and resistance to bending and cyclic fatigue of ProTaper Gold and ProTaper Universal instruments. J Endod 2015;41:1134–8.
- Silva EJ, Muniz BL, Pires F, et al. Comparison of canal transportation in simulated curved canals prepared with ProTaper Universal and ProTaper Gold systems. Restor Dent Endod 2016;41:1–5.
- De-Deus G, Moreira EJ, Lopes HP, Elias CN. Extended cyclic fatigue life of F2 Pro-Taper instruments used in reciprocating movement. Int Endod J 2010;43:1063–8.
- Pedullà E, Grande NM, Plotino G, et al. Influence of continuous or reciprocating motion on cyclic fatigue resistance of 4 different nickel-titanium rotary instruments. J Endod 2013;39:258–61.
- Kiefner P, Ban M, De-Deus G. Is the reciprocating movement per se able to improve the cyclic fatigue resistance of instruments? Int Endod J 2014;47:430–6.
- ISO 3630-1:2008. Dentistry root canal instruments part 1: general requirements and test methods. Geneva, Switzerland: International Organization for Standardization; 2008.
- De-Deus G, Leal Vieira VT, Nogueira da Silva EJ, et al. Bending resistance and dynamic and static cyclic fatigue life of Reciproc and WaveOne large instruments. J Endod 2014;40:575–9.

- ANSI/ADA. Specification no. 28. Root canal files and reamers, type K for band use. Chicago: American Dental Association; 2002.
- Grande NM, Plotino G, Pecci R, et al. Cyclic fatigue resistance and three-dimensional analysis of instruments from two nickel-titanium rotary systems. Int Endod J 2006; 39:755–63
- Plotino G, Grande NM, Sorci E, et al. A comparison of cyclic fatigue between used and new Mtwo Ni-Ti rotary instruments. Int Endod J 2006;39:716–23.
- Plotino G, Grande NM, Sorci E, et al. Influence of a brushing working motion on the fatigue life of NiTi rotary instruments. Int Endod J 2007;40:45–51.
- Plotino G, Grande NM, Melo MC, et al. Cyclic fatigue of Ni–Ti rotary instruments in a simulated apical abrupt curvature. Int Endod J 2010;43:226–30.
- Zinelis S, Eliades T, Eliades G. A metallurgical characterization of tem endodontic Ni-Ti instruments: assessing the clinical relevance of shape memory and superelastic properties of Ni-Ti endodontic instruments. Int Endod J 2010;43:125–34.
- Shen Y, Coli JM, Zhou H, et al. HyFlex nickel-titanium rotary instruments after clinical use: metallurgical properties. Int Endod J 2013;46:720–9.
- Gao Y, Gutmann JL, Wilkinson K, et al. Evaluation of the impact of raw materials on the fatigue and mechanical properties of ProFile Vortex rotary instruments. J Endod 2012;38:398–401.