International Endodontic Journal

doi:10.1111/j.1365-2591.2010.01835.x

Mechanical properties of nickel–titanium rotary instruments produced with a new manufacturing technique

G. Gambarini[1], G. Plotino[1], N. M. Grande[1], D. Al-Sudani[2], M. De Luca[1] & L. Testarelli[1]

[1]Department of Endodontics, 'Sapienza' University of Rome, Rome, Italy; and [2]Department of Endodontics, KSU University, Riyadh, Saudi Arabia

Abstract

Gambarini G, Plotino G, Grande NM, Al-Sudani D, De Luca M, Testarelli L. Mechanical properties of nickel–titanium rotary instruments produced with a new manufacturing technique. *International Endodontic Journal*, **44**, 337–341, 2011.

Aim To investigate whether flexibility and cyclic fatigue resistance was increased for nickel–titanium instruments produced by a new manufacturing technique.

Methodology Forty K3 tip size 25, 0.06 taper (SybronEndo) nickel–titanium rotary instruments were randomly selected and divided into two groups ($n = 20$). One group served as control, being the commercially available instruments produced with a traditional grinding process (K3). The second group of instruments (K4 prototypes) were then subjected to a proprietary thermal treatment after the grinding process. Finally, each group was randomly divided into two subgroups of 10 instruments each, to perform the stiffness test and the cyclic fatigue test. All data were recorded and subjected to statistical evaluation using Student's *t*-test. Significance was set at the 95% confidence level.

Results For the stiffness test, a statistically significant difference ($P < 0.05$) was noted between K3 and K4 prototype instruments. K4 prototype instruments were significantly more flexible when compared to K3 instruments (59.3 ± 4.3 vs. 98.1 ± 6.4 g cm^{-1}). For the cyclic fatigue test, a significant difference ($P < 0.05$) was noted between K3 and K4 prototype instruments. K4 prototype instruments demonstrated a significant increase in the mean number of cycles to failure (NCF) when compared to K3 instruments (1198 ± 279 vs. 542 ± 81 NCF).

Conclusions The new manufacturing technique resulted in the K4 prototype instruments having enhanced mechanical properties, compared to K3 instruments, manufactured with a traditional grinding process.

Keywords: cyclic fatigue, endodontic instruments, flexibility, nickel–titanium, thermal treatment.

Received 8 September 2010; accepted 7 November 2010

Introduction

One of the major innovations in endodontics has been the introduction of nickel–titanium (NiTi) alloy to manufacture root canal instruments. This is mainly because of the superelasticity of the NiTi alloy, which provides increased flexibility and allows the instruments to effectively follow the original path of the root canal (Thompson 2000). Unfortunately, bending properties are limited by the size and taper of instruments. As instruments become larger in tip size or taper, there is a progressive reduction in the flexibility they possess, which increases the risk of zips, ledges and canal transportation (Melo *et al.* 2008). Moreover, instruments with larger diameters have been found to

Correspondence: Gianluca Plotino, DDS, PhD, Department of Endodontics, 'Sapienza' University of Rome, Via Tommaso Salvini, 57 – 00197 Rome, Italy (Tel.: +393396910098; e-mail: endo@gianlucaplotino.com).

succumb to flexural fatigue earlier than those with smaller diameters (Pruett *et al.* 1997, Haikel *et al.* 1999), and they appear to have greater internal stress accumulation (Ullmann & Peters 2005). On the contrary, an increase in instrument diameter and corresponding increase in cross-sectional area may contribute to increased resistance to torsional failure (Parashos & Messer 2006).

All these properties play a major role in determining performance and safety of nickel–titanium rotary instrumentation. Several studies demonstrated that fracture of nickel–titanium instruments used in rotary motion occurs in two different ways: fracture because of torsion and fracture because of flexural fatigue (Serene *et al.* 1995, Sattapan *et al.* 2000, Ullmann & Peters 2005). Torsional fracture occurs when an instrument tip or another part of the instrument is locked in a canal whilst the shank continues to rotate. When the elastic limit of the metal is exceeded by the torque exerted by the handpiece, fracture of the tip becomes inevitable (Martín *et al.* 2003). Fracture because of fatigue through flexure occurs because of metal fatigue. The instrument does not bind in the canal but it rotates freely in a curvature, generating tension/compression cycles at the point of maximum flexure until the fracture occurs (Peters 2004).

Possible strategies to increase efficiency and safety of nickel–titanium (NiTi) rotary include an improvement in the manufacturing process, or the use of new alloys that provide superior mechanical properties. A new NiTi alloy, termed the M-Wire™ (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) was developed in 2007. Before the grinding process, the alloy is thermally treated to improve its properties. The final goal is to produce instruments with greater flexibility and increased resistance to cyclic fatigue, compared to those constructed from traditional NiTi alloy (Larsen *et al.* 2009). In the year 2008, a completely different manufacturing process was developed by SybronEndo (Orange, CA, USA), to create a new nickel–titanium rotary file for root canal preparation, called the Twisted File ™ (TF™). It utilizes twisting of a ground blank in combination with a complex heating and cooling treatment, to reportedly enhance superelasticity and increase cyclic fatigue resistance (Gambarini *et al.* 2008). As all the commercially available instruments manufactured with the above-mentioned manufacturing processes (TF™, GTX™, Vortex™) have different and innovative designs, it is not easy to determine precisely whether, or to which extent, the improvements in the mechanical properties are related to their new designs or to the manufacturing process.

More recently, a new manufacturing process has been introduced by SybronEndo, as a development of TF technology. More precisely, a special thermal process (patent pending) is applied to nickel–titanium endodontic instruments after the grinding process is completed, aiming to transform the alloy into a slightly different phase of crystalline structure. Theoretically, the main advantage is not only to improve flexibility and strength with a thermal treatment, but at the same time to accommodate some of the internal stress caused by the grinding process, by modifying the crystalline structure of the alloy. By doing so, the new technique could eliminate many drawbacks of the grinding process and produce instruments with a superior mechanical resistance.

As there have been no studies published to date on the mechanical properties of instruments produced by this new manufacturing technique, the aim of this study is to investigate whether flexibility and cyclic fatigue resistance is increased for nickel–titanium instruments manufactured using this new process. This was evaluated by comparing K3 instruments and prototype instruments, K4, (SybronEndo). As all tested instruments had the same dimensions and design, were produced using the same raw material and grinding process, any change in their mechanical properties could be attributed only to the new technology, the proprietary thermal treatment performed on K4 prototype instruments after the grinding process.

Materials and methods

Forty K3 (SybronEndo) nickel–titanium rotary instruments from the same production batch were selected randomly and divided into two groups ($n = 20$). All the instruments were produced with the same raw material, the same grinding process and had the same design and dimensions of the tip (0.25) and taper (0.06). One group served as control, being the commercially available instruments produced with a traditional grinding process (K3). The second group of instruments (K4 prototypes) were then subjected to a proprietary thermal treatment after the grinding process. Finally, each group was randomly divide into two subgroups of ten instruments each, to perform the stiffness test ($n = 10$) and the cyclic fatigue test ($n = 10$).

For the stiffness test, testing procedures strictly followed guidelines published in ISO standard 3630-1. Bending moment was measured when the instrument attained a 45° bend. Each instrument was inserted in a chuck connected to an electric motor revolving at 2.0 rotations per second (rpm). Three millimetres of the tip were clamped in a chuck connected to a digital torque meter memocouple. The amplifier was set at an angular deflection of 45°, at which point the test stopped automatically. The bending moment was then measured and recorded by the memocouple connected to a computer. All data were recorded and subjected to statistical evaluation using Student's *t*-test. Significance was set at the 95% confidence level.

The cyclic fatigue testing device used in this study has been utilized for studies on cyclic fatigue resistance previously performed by the authors (Grande *et al.* 2006, Plotino *et al.* 2006). The device consists of a main frame to which a mobile plastic support is connected for the electric handpiece, and a stainless-steel block containing the artificial canals. The electric handpiece was mounted upon a mobile device to allow precise and reproducible placement of each instrument inside the artificial canal. This ensured three-dimensional alignment, and positioning of the instruments to the same depth. The artificial canal was manufactured by reproducing an instrument's size and taper, thus providing the instrument with a suitable trajectory that respect the parameters of the curvature chosen. A simulated root canal with a 60° angle of curvature and 5 mm radius of curvature was constructed for each instrument. The centre of the curvature was 6 mm from the tip of the instrument, and the curved segment of the canal was approximately 6 mm in length. The instruments were rotated at a constant speed of 300 rpm using a 16 : 1 reduction handpiece (W&H Dentalwerk, Burmoos, Austria), powered by a torque controlled electric motor (X-Smart; Dentsply Maillefer, Ballaigues, Switzerland). To reduce the friction of the file as it contacted the artificial canal walls, a special high-flow synthetic oil designed for lubrication of mechanical parts (Super Oil; Singer Co. Ltd, Elizabethport, NJ, USA) was applied.

All instruments were rotated until fracture occurred. The time to fracture was recorded visually with a 1/100-second chronometer. The time to fracture recorded in seconds was multiplied by the number of rotations per second (RPM/60), to obtain the number of cycles to failure (NCF) for each instrument. The length of the fractured tip was also recorded for each instrument.

Means and standard deviations of NCF and fragment length were calculated for each system. Data were subjected to Student's *t*-test to determine significant differences between groups for both variables considered. Significance was set at the 95% confidence level.

Table 1 Mean values ± standard deviation of the values obtained in the stiffness test

K4 prototypes (g cm^{-1})	59.3* ± 4.3
K3 (g cm^{-1})	98.1 ± 6.4

*Significant difference ($P < 0.05$).

Results

For the stiffness test, mean values ± standard deviation expressed as gram per centimetre are displayed in Table 1. A higher value is attributed to a higher rigidity of the instruments. A statistically significant difference ($P < 0.05$) was noted between K3 and K4 prototype instruments (Table 1). K4 prototype instruments showed a significant increase in flexibility when compared to K3 instruments (59.3 ± 4.3 vs. 98.1 ± 6.4 g cm^{-1}).

For the cyclic fatigue test, mean values ± standard deviation expressed as NCF are displayed in Table 2. A higher NCF is attributed to a higher resistance to cyclic fatigue of the tested instruments. A statistically significant difference ($P < 0.05$) was noted between K3 and K4 prototype instruments (Table 2). K4 prototype instruments showed a significant increase in the mean NCF when compared to K3 instruments (1198 ± 279 vs. 542 ± 81 NCF).

Mean length of the fractured segment was also recorded to evaluate the correct positioning of the tested instrument inside the canal curvature and whether similar stresses were being induced. No statistically significant difference ($P > 0.05$) in the mean length of the fractured fragments was evident for all of the instruments (Table 2).

Discussion

ANSI/ADA Specification No. 28 (2002) prescribes tests to measure strength under torsion and flexibility of stainless-steel hand files. The same tests are adopted by

Table 2 Mean values ± standard deviation of the number of cycles to failure (NCF) and the fragment length (FL) obtained in the cyclic fatigue test

	NCF	FL (mm)
K4 prototypes	1198* ± 279	5.8 ± 0.3
K3	542 ± 81	5.7 ± 0.4

*Significant difference ($P < 0.05$).

ISO 3630/1 (2008), which is designed for instruments having 0.02 ISO taper. To date, there is no specification or International Standard to test cyclic fatigue resistance of nickel–titanium endodontic rotary instruments of greater taper, although the ISO and ADA are currently working to develop a new standard for these instruments. In this study, flexibility was evaluated strictly following procedures described by ISO 3630-1, whilst cyclic fatigue tests were performed using a methodology introduced by the authors and validated in many studies published in peer-reviewed journals (Grande *et al.* 2006, Plotino *et al.* 2006, 2007, 2009, 2010, Gambarini *et al.* 2008).

Results demonstrated that the new manufacturing technique produced K4 prototype instruments with enhanced mechanical properties, compared to K3 instruments, manufactured with a traditional grinding process. The improvement was highly significant, with the prototype instruments being approximately two times more flexible and more resistant to cyclic fatigue. Because the raw material, the grinding process and the instruments design and dimensions were the same, the new technology used for the fabrication of the tested K4 prototypes could be the only possible explanation of this improvement.

Nickel–titanium is a shape memory alloy that is superelastic and when stressed during canal instrumentation undergoes a molecular phase transformation. It was demonstrated that the mechanical properties and various phase transformation of the NiTi alloy are dependent on thermo-mechanical processing (Kuhn *et al.* 2001). Superelastic properties of nickel–titanium alloys are influenced by numerous factors, including changes in its composition, machining characteristics and differences in heat treatment (Thompson 2000). Miyazaki *et al.* (1982) found superelasticity of NiTi alloys to be greatly dependent on the thermal history of the material. They showed various heat treatments can produce or eliminate superelasticity behaviour. In a recent study Hayashi *et al.* (2007) stated that additional heat treatment of nickel–titanium instruments may be effective in increasing the flexibility of nickel–titanium rotary instruments. The improvement could be attributed to an increase in the proportion of martensite (which is known to be more flexible than austenitic NiTi) within the material as a result of the heat treatment.

Improvements in the mechanical properties of the alloy could also be related to partial annihilation of lattice defects that occur when the alloy is thermally treated. When the material is subjected to deformation or stress by machining a high density of lattice defects is produced as dislocations. When the metal is heated up, a recrystallization process can take place, decreasing the density of lattice defects and internal stress produced by work hardening. This should improve the flexibility and strength of the alloy. Other authors reported that thermal treatments at 400 °C before machining were able to reduce defects in the alloy caused by work hardening, which could disturb the phase transformation (Kuhn & Jordan 2002). It is theoretically possible that thermal treatments after machining can do the same and reduce defects in the alloy caused by the grinding process.

The results of this study could confirm these hypotheses. The post-grinding heat treatment seems not only to improve flexibility of nickel–titanium rotary instruments, but to eliminate many drawbacks of the grinding process and provide a superior mechanical resistance, as well. The clinical advantages of the new manufacturing process could theoretically be relevant. Such a significant increase in material flexibility to construct NiTi instruments of increased taper should allow preparation of curved canals with less risk of canal transportation and iatrogenic errors. Such a significant increase in material resistance to fatigue should reduce the risk of intracanal breakage. It is clear that the positive findings of this study must be confirmed by other *in vitro* and *in vivo* investigations.

Acknowledgements

The authors thank SybronEndo for providing K3 instruments and K4 prototypes. The study was supported by a grant from SybronEndo.

References

ANSI/ADA Specification N° 28 (2002) . *Root Canal Files and Reamers, Type K for Hand Use*. Chicago: American Dental Association.

Gambarini G, Grande NM, Plotino G *et al.* (2008) Fatigue resistance of engine-driven rotary nickel–titanium instruments produced by new manufacturing methods. *Journal of Endodontics* **34**, 1003–5.

Grande NM, Plotino G, Pecci R, Bedini R, Malagnino VA, Somma F (2006) Cyclic fatigue resistance and three-dimensional analysis of instruments from two nickel–titanium rotary systems. *International Endodontic Journal* **39**, 755–63.

Haikel Y, Serfaty R, Bateman G, Senger B, Allemann C (1999) Dynamic and cyclic fatigue of engine-driven rotary nickel titanium endodontic instruments. *Journal of Endodontics* **25**, 434–40.

Hayashi Y, Yoneyama T, Yahata Y *et al.* (2007) Phase transformation behaviour and bending properties of hybrid nickel–titanium rotary endodontic instruments. *International Endodontic Journal* **40**, 247–53.

ISO 3630-1 (2008) . Dentistry – root canal instruments – Part 1: general requirements and test methods.

Kuhn G, Jordan L (2002) Fatigue and mechanical properties of nickel–titanium endodontic instruments. *Journal of Endodontics* **28**, 716–20.

Kuhn G, Tavernier B, Jordan L (2001) Influence of structure on nickel–titanium endodontic instruments failure. *Journal of Endodontics* **27**, 516–20.

Larsen CM, Watanabe I, Glickman GN, He J (2009) Cyclic fatigue analysis of a new generation of nickel titanium rotary instruments. *Journal of Endodontics* **35**, 401–3.

Martín B, Zelada G, Varela P *et al.* (2003) Factors influencing the fracture of nickel titanium rotary instruments. *International Endodontic Journal* **36**, 262–6.

Melo MCC, Pereira ESJ, Viana ACD, Fonseca AMA, Buono VTL, Bahia MGA (2008) Dimensional characterization and mechanical behaviour of K3 rotary instruments. *International Endodontic Journal* **41**, 329–38.

Miyazaki S, Ohmi Y, Otsuka K, Suzuki Y (1982) Characteristics of deformation and transformation pseudoelasticity in NiTi alloys. *Journal de Physique* **15**, 287–92.

Parashos P, Messer HH (2006) Rotary NiTi instrument fracture and its consequences. *Journal of Endodontics* **32**, 1031–43.

Peters OA (2004) Current challenges and concepts in the preparation of root canal systems: a review. *Journal of Endodontics* **30**, 559–65.

Plotino G, Grande NM, Sorci E, Malagnino VA, Somma F (2006) A comparison of cyclic fatigue between used and new Mtwo Ni-Ti rotary instruments. *International Endodontic Journal* **39**, 716–23.

Plotino G, Grande NM, Sorci E, Malagnino VA, Somma F (2007) Influence of a brushing working motion on the fatigue life of NiTi rotary instruments. *International Endodontic Journal* **40**, 45–51.

Plotino G, Grande NM, Cordaro M, Testarelli L, Gambarini G (2009) Measurement of the trajectory of different NiTi rotary instruments in an artificial canal specifically designed for cyclic fatigue tests. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology and Endodontology* **108**, e152–6.

Plotino G, Grande NM, Melo MC, Bahia MG, Testarelli L, Gambarini G (2010) Cyclic fatigue of Ni–Ti rotary instruments in a simulated apical abrupt curvature. *International Endodontic Journal* **43**, 226–30.

Pruett JP, Clement DJ, Carnes DL (1997) Cyclic fatigue testing of nickel titanium endodontic instruments. *Journal of Endodontics* **23**, 77–85.

Sattapan B, Nervo GJ, Palamara JEA, Messer HH (2000) Defects in rotary nickel–titanium files after clinical use. *Journal of Endodontics* **26**, 161–5.

Serene TP, Adams JD, Saxena A (1995). *Nickel–Titanium Instruments. Applications in Endodontics*. St Louis, MO: Ishiyaku EuroAmerica, Inc.

Thompson SA (2000) An overview of nickel–titanium alloys used in dentistry. *International Endodontic Journal* **33**, 297–310.

Ullmann CJ, Peters OA (2005) Effect of cyclic fatigue on static fracture loads in ProTaper nickel–titanium rotary instruments. *Journal of Endodontics* **31**, 183–6.