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Influence of sterilization on mechanical properties and fatigue resistance of nickel–titanium rotary endodontic instruments

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Abstract

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Aim To evaluate the effect of repeated sterilization cycles in dry oven or autoclave, on the mechanical behaviour and fatigue resistance of rotary endodontic Ni–Ti instruments.

Methodology New Ni–Ti instruments were subjected to five consecutive sterilization cycles in a dry oven or steam autoclave. Microhardness was measured in the nonmachined parts of the shanks of instruments using a Vickers indenter. Specimens of Ni–Ti wires were submitted to the same sterilization protocol and tensile tested until rupture. A group of instruments were fatigued to one half of their average fatigue life and then sterilized. New and sterilized instruments were fatigue tested until rupture. ANOVA tests at $\alpha = 0.05$ were used for statistical analysis.

Results Sterilization procedures resulted in no significant changes in Vickers microhardness, nor in the parameters describing the mechanical behaviour of the wires. However, the number of cycles to failure was statistically higher for all instruments after dry heat or autoclave sterilization cycles. In the instruments previously fatigued to one half of their fatigue life, autoclave sterilization gave rise to an increase of 39% in the remaining number of cycles to failure.

Conclusions Changes in the mechanical properties of Ni–Ti endodontic instruments after five cycles of commonly used sterilization procedures were insignificant. The sterilization procedures are safe as they produced a significant increase in the fatigue resistance of the instruments.

Keywords: endodontic instruments, fatigue resistance, microhardness, nickel–titanium, sterilization, tensile properties.

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Introduction

Infection control is a major issue in dentistry, mainly because of the concern over contagious diseases transmitted in healthcare settings. The cleaning and sterilization of endodontic instruments between treatment sessions are essential to prevent cross infection. However, the effect of the heating and cooling cycles employed during sterilization on the mechanical properties and resistance to fracture of endodontic instruments have not yet been clearly stated. For instance, Mitchell *et al.* (1983) observed a reduction in the values of angular deflection of stainless steel files after 10 sterilization cycles in steam autoclave, but these results were not confirmed by Iverson *et al.* (1985), who submitted two different kinds of stainless steel endodontic instruments to 10 cycles of autoclaving, dry heat and cold chemical sterilization and verified that their torsional strength was not affected by the sterilization procedures.

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Reports on the effects of sterilization upon nickel–titanium (Ni–Ti) rotary endodontic instruments also do not clarify the subject. Silvaggio & Hicks (1997) demonstrated that sterilization, either in autoclave or in dry heat, did not reduce the resistance of Ni–Ti instruments. In turn, Mize *et al.* (1998), after sterilizing Ni–Ti instruments cycled to 25%, 50% and 75% of their average fatigue life, showed that no significant difference in fatigue resistance had taken place. Hilt *et al.* (2000), comparing instruments made of stainless steel and Ni–Ti submitted to sterilization in autoclave or dry heat, showed that the torsional strength, hardness and microstructure of these instruments was not affected by the number of cycles nor by the process of sterilization. Canalda-Shali *et al.* (1998), using a similar methodology, reported a reduction in the torsional strength of stainless steel files, and nonconclusive results for Ni–Ti instruments. The same study reported a reduction in the flexibility of both types of instruments after sterilization. According to Serene *et al.* (1995), the deformation suffered by Ni–Ti instruments during clinical use can be recovered by heating them to a temperature above 125 °C. The same authors verified that the Vickers microhardness increased approximately 18% for Ni–Ti wires after one and five cycles of sterilization in dry heat or autoclave. Melo *et al.* (2002) also observed an increase of 10% in the average values of Vickers microhardness of Ni–Ti endodontic instruments after five sterilization cycles.

The approximately equiatomic Ni–Ti alloy used in the manufacture of rotary endodontic instruments exhibits two special characteristics: the shape memory effect (SME) and superelasticity (SE), in which the metal, deformed in an apparently permanent way, recovers its original shape when heated – SME, or after the removal of the stress – SE. Both effects in Ni–Ti alloys are related to the transformation of austenite, the β-phase, with the B2 cubic crystal structure, to martensite, with the monoclinic B19' crystal structure. This phase transformation is thermoelastic, that is, can be induced by cooling or by the application of stress. When the metal is heated (SME) or the stress is released (SE), the reverse transformation, martensite to austenite, takes place. The lower symmetry of martensite in relation to austenite makes the reverse transformation to undo the previously induced shape change causing shape recovery (Otsuka & Wayman 1998).

The mechanical properties and the transformation temperatures of Ni–Ti alloys are strongly dependent on their chemical composition and thermomechanical processing. Usually, superelastic Ni–Ti alloys contain 56 weight% Ni and 44 weight% Ti (Thompson 2000). This composition is almost equiatomic, but has a small amount of Ni in excess of 50 atomic%. Ageing these alloys above 350 °C causes precipitation of the intermediate phase Ti_3Ni_4, in the form of submicrometer particles, giving rise to precipitation hardening of the β-phase (Saburi 1998). Precipitation hardening of austenite is a well-known method used to improve SME and SE shape recovery capacity (Miyazaki *et al.* 1982, Saburi *et al.* 1982).

Despite the increased flexibility of engine-driven Ni–Ti endodontic instruments, their continuous rotation within curved root canals causes structural fatigue as a result of bending and torsional stresses (Sotokawa 1988, Pruett *et al.* 1997). The geometry of the canals, described by the radius and angle of curvature, associated with the diameter of the instrument in the region of maximum bending (given by the distance of the curvature to the instrument tip) determines the maximum tensile strain amplitude on the instrument shank and thus its fatigue life (Bahia & Buono 2005). Therefore, the resistance of rotary instruments to breakage because of fatigue decreases as their diameter increases and the curvature radius of the canal is reduced.

This study was performed with the objective of clarifying the effects of sterilization on the mechanical behaviour and fatigue resistance of rotary Ni–Ti endodontic instruments. Sterilization was carried out in dry heat oven and steam autoclave in new ProFile instruments (Dentsply Maillefer, Ballaigues, Switzerland). The influence of sterilization in new instruments previously cycled to one-half of their fatigue life was also evaluated.

Materials and methods

New ProFile Ni–Ti endodontics instruments (Dentsply Maillefer) were submitted to five consecutive cycles of sterilization in a dry heat oven (Olidef, Ribeirão Preto, SP, Brazil) or in a steam autoclave (Cristófoli, Campo Mourão, PR, Brazil). Dry heat sterilization cycles include heating to 170 °C in 40 min, holding at this temperature for 60 min, and then cooling to room temperature in 55 min. Distilled water was used in steam sterilization, which was performed at a pressure between 1.4 and 1.8 kgf cm^{-2}, in the temperature interval of 122–128 °C, within a total sterilization time of 64 min (heating in 10 min, sterilization in 21 min and drying in 33 min). Instruments were placed in glass vials and properly conditioned in a stainless steel box for dry heat sterilization or in autoclavable Steribag® bags (SSWhite, Rio de Janeiro, RJ, Brazil). Sterilization was monitored by using the chemical indicator tape 3M® (Sumaré, SP, Brazil) in each cycle.

Measurements of Vickers microhardness were carried out on the nonmachined part of the shanks of size 30, 0.06 taper ProFile instruments, in a microhardness tester (FM-1, Future-Tech, Tokyo, Japan). Thirty new instruments and 60 instruments sterilized in dry heat or autoclave, 30 instruments for each condition, were employed to obtain these measurements. All instruments had their handles removed before being placed in a grooved support, and three indentations, with a load of 0.2 kgf, were made on each instrument shaft. The ANOVA general linear model test was employed to evaluate the statistical significance of the results.

Specimens of Ni–Ti wires employed in the manufacture of ProFile rotary endodontic instruments were provided by Dentsply Maillefer (Ballaigues, Switzerland). Wires with 1.2-mm diameter and 100-mm length were tensile tested until rupture in a universal testing machine (Instron 5581, Canton, MA, USA). A slow cross-head speed of 1.5 mm min^{-1}, corresponding to a strain rate of 1.0×10^{-3} s^{-1}, was employed, with an extensometer of 25-mm length, in order to determine the parameters describing the mechanical behavior of the wires. These parameters are the transformation stress, which is the value of the stress corresponding to the end of the elastic portion of the stress–strain curve in a superelastic material, associated with the start of transformation of austenite to martensite; the stress at maximum load, generally called the ultimate tensile strength, which is the maximum stress the specimen can withstand before rupture; and the plastic strain at breakage, called the total elongation, which is the total permanent deformation imposed in the test, expressed as a percentage of the initial gauge length, which is the extensometer length of 25 mm. The tensile tests were carried out in wire samples in the as-received condition and in samples previously submitted to five consecutive sterilization cycles as described before (three specimens for each condition). The results were determined as the average of the three tests, using the software INSTRON SERIES IX for Windows®.

Fatigue tests were carried out in a bench device that allowed the files to rotate freely inside an artificial canal made of AISI H13 tool steel, consisting of an arch with a curvature angle of 45°, a radius of 5 mm, and a guide cylinder of 10-mm diameter, made of the same material (Bahia & Buono 2005). The chosen geometry placed the area of maximum tensile strain amplitude at about 3 mm from the tip of the instrument. After machining, the artificial canal was quenched, to prevent wear by friction with the rotating instruments, which was minimized by the use of a mineral oil as lubricant. The rotation speed was 250 rpm, applied by an endodontic electric motor (Endo-Plus, VK-Driller, São Paulo, SP, Brazil), operating with a torque of 5 Ncm^{-1}, with a hand piece of 16 : 1 reduction (WH 975 DentalWork, Burmoos, Austria). The number of cycles to failure (NCF) was obtained by multiplying the rotation speed employed in the fatigue test device by the test time, registered with a digital chronometer. The point of fracture in relation to the tip of the instrument was determined by measuring the fractured instrument with an endodontic ruler.

Twenty new ProFile instruments, sizes 25 and 30, 0.06 taper (10 of each type), were tested until rupture in the fatigue test device described, to establish their fatigue life by means of the average NCF. Following that, 30 new ProFile instruments sizes 25 and 30, 0.06 taper (15 of each type), previously submitted to five consecutive sterilization cycles in dry heat or autoclave, were tested in the same device, in order to assess the influence of the sterilization processes on their fatigue life. Another 15 size 30, 0.06 taper instruments, previously cycled to one half of their fatigue life and then sterilized in an autoclave, were also tested in the same device until rupture, in order to evaluate the influence of the sterilization processes on the fatigue life of an used instrument. One-way ANOVA tests at $\alpha = 0.05$ were employed to evaluate the statistical significance of the measured values.

Results

The average values (and SD) for the Vickers microhardness measurements for nonsterilized ProFile instruments and for those sterilized by dry heat or steam autoclave were 361 (39), 368 (37) and 365 (30), respectively. Although a small increase in absolute mean values occurred for sterilized instruments, there was no statistically significant difference between them and the nonsterilized instruments. Similarly, no difference was found in Vickers microhardness amongst instruments sterilized by the two processes employed.

The results of the tensile tests performed on the Ni–Ti wires employed in the manufacture of ProFile endodontic instruments are summarized in Table 1, which shows mean values and SD of the transformation stress, ultimate tensile strength and plastic strain at breakage,

Table 1 Average values (and SD) of transformation stress, stress at maximum load and plastic strain at breakage

Condition	Transformation stress (MPa)	Stress at maximum load (MPa)	Plastic strain at breakage (%)
As received	533 (10)	1362 (70)	11.9 (0.8)
Dry heat	543 (06)	1420 (10)	12.5 (0.4)
Autoclave	530 (05)	1421 (05)	12.9 (0.1)

determined for the nonsterilized and sterilized wires. The average tensile stress–strain curves obtained in these tests are shown in Fig. 1.

The average NCF and SD determined in the fatigue tests of the instruments are shown in Table 2. The fatigue resistance of Ni–Ti ProFile instruments, measured by the NCF values, shows a tendency to decrease as the size of the instruments increases. The statistical analysis of the average values of NCF for sizes 25 and 30, 0.06 taper, nonsterilized and sterilized instruments showed significant differences amongst them ($P < 0.05$), pointing to the fact that sterilization increased their fatigue resistance. For size 25, 0.06 taper instruments, the average values of NCF increased 14% and 16% after sterilization in dry heat and steam autoclave, respectively. For size 30, 0.06 taper instruments, this increase was 26% for dry heat and 27% for autoclave sterilization. On the other hand, when a comparative statistical analysis using one-way ANOVA tests was made amongst instruments sterilized by the two procedures, no significant difference ($P \geq 0.05$) was found in the NCF values. Moreover, in all combinations tested, no statistically significant difference ($P \geq 0.05$) was observed amongst instrument fracture position. The average values of this parameter (and SD) were 22.2 (0.4) mm, 21.9 (0.5) mm and 22.0 (0.7) mm, for nonsterilized, dry heat and autoclave sterilized instruments, respectively.

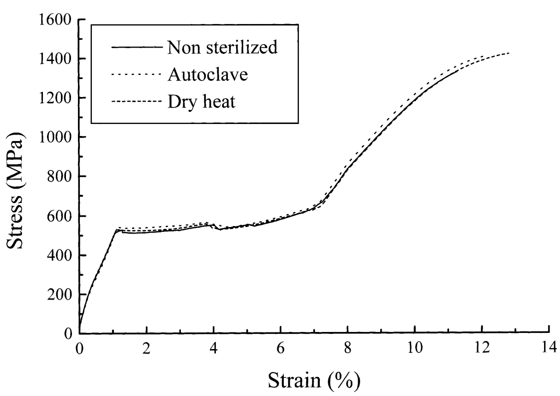

Figure 1 Average stress–strain curves obtained in tensile tests of Ni–Ti wires employed in the manufacture of ProFile instruments.

Table 2 Average values (and SD) of the number of cycles to failure

Condition	Instrument 25/0.06	Instrument 30/0.06
As received	790 (82)	712 (160)
Dry heat	901 (105)	894 (210)
Autoclave	919 (136)	950 (127)

The average remaining cycles for size 30, 0.06 taper instruments previously cycled to one-half of their fatigue life was 352 (85) and, after five cycles of sterilization in autoclave, 493 (157). A one-way ANOVA test showed a statistically significant ($P < 0.05$) increase in the remaining NCF of sterilized instruments. The increase in the average values of remaining cycles was 38.5%.

Discussion

Dental professionals are exposed to a wide variety of microorganisms in the blood and saliva of patients, which may cause infectious diseases such as the common cold, pneumonia, tuberculosis, herpes, hepatitis B, C and acquired immune deficiency syndrome (AIDS). The use of effective infection control procedures and universal precautions in the dental surgery will prevent cross-contamination. Aseptic techniques are especially important in endodontics because microorganisms are the major cause of endodontic disease. Sterilization of endodontic instruments is important for two reasons: the elimination of patient cross-contamination and the increase in the success of the endodontic therapy (Hurtt & Rossman 1996). The ADA Council on Dental Therapeutics, Council on Dental Practice (1988, 1996) has recommended that heat sterilization be used for all instruments that can withstand repeated exposure to the required sterilization temperature. Steam autoclave, dry heat oven and ethylene oxide are said to be the most effective and preferred methods for the sterilization of endodontic instruments. In the present work, sterilization in dry heat oven and steam autoclave were chosen because they are the most accessible methods.

One of the problems presented by stainless steel endodontic instruments is the corrosion caused by sterilization in conventional steam autoclaves. Thus, dry heat sterilization of these instruments is preferred by many professionals. However, dry heat is not as penetrating as steam autoclave sterilization, and also demands higher temperatures and longer times (Reams *et al.* 1995, Miller 1996). It is clear that the physical properties of endodontic instruments, such as cutting efficiency, flexibility, resistance to torsional and fatigue fracture, should not be significantly altered by the sterilization procedures (Canalda-Shali *et al.* 1998). The choice of five sterilization cycles was based on literature reports stating that rotary Ni–Ti instruments can be safely utilized to shape 10 curved root canals (Yared *et al.* 1999, Gambarini 2001). As only curved root canals induce fatigue in the instruments, five cycles of sterilization correspond to clinical use during the average useful life of the instrument. Because the main interest was to detect possible cumulative changes in properties associated with the heating and cooling cycles, they were applied consecutively to the new and the previously fatigued instruments. The instruments sizes 25 and 30, 0.06 taper, were chosen because they present the lowest fatigue resistance amongst ProFile instruments (Bahia & Buono 2005).

As pointed out by Thompson (2000), Ni–Ti endodontic instruments are machined from wires containing about 56 weight% Ni, which are previously cold drawn and then annealed in the temperature range which favours partial recovery of dislocations and precipitation of Ti_3Ni_4. After the machining process, no thermal treatment is applied to the Ni–Ti endodontic instruments (Thompson 2000). From the metallurgical point of view, the temperature used in sterilization may not be high enough to cause significant changes in the alloy structure. The required changes to increase hardness, cutting efficiency and fatigue life would be related to precipitation of Ti_3Ni_4 particles (Khalil-Allafi *et al.* 2004, Otsuka & Ren 2005). However, it is possible that consecutive cycles of sterilization give rise to cumulative effects, leading to increases in hardness in rotary Ni–Ti endodontic instruments after sterilization, as observed by Serene *et al.* (1995) and Melo *et al.* (2002), in Ni–Ti wires and instruments, respectively. On the other hand, Hilt *et al.* (2000) did not observe this effect on similar instruments.

The present study showed a small increase in hardness of Ni–Ti ProFile instruments after five consecutive sterilization cycles, but, as mentioned previously, this increase could not be statistically confirmed. What can be stated is that even if sterilization does not render rotary Ni–Ti endodontic instruments stronger, it does not result in deleterious effects that could reduce their mechanical resistance. This is an important result in itself, implying that sterilization, either in dry heat or in steam autoclaves, does not compromise the mechanical behaviour of rotary Ni–Ti endodontic instruments, assuring the possibility of their re-use after sterilization.

The tensile stress–strain curves showed in Fig. 1 disclose important characteristics of the behaviour of Ni–Ti superelastic alloys. The stress peak at the beginning of the superelastic plateau corresponds to the nucleation of martensite variants in austenite, whilst the subsequent decrease in stress occurs because the propagation of these convenient-orientated martensite variants requires lower stresses (Huang & Liu 2001). Comparison of the parameters describing the mechanical behaviour of the Ni–Ti wires (Table 1, Fig. 1) indicates that this behaviour is not influenced by sterilization. This result is in agreement with those obtained in the Vickers microhardness tests and confirms that no change in the bulk properties of the material are induced by sterilization.

In view of this, the observed increase in fatigue resistance associated with the two sterilization methods deserves a more detailed examination. Fatigue strength is one of the most important aspects to be considered in device applications using rotary parts. The term fatigue is appropriate, as it refers to the time-delayed failure of materials subjected to cyclical stress and involves three stages: crack nucleation, slow and gradual growth, and fast final fracture. Nucleated cracks grow as a result of a time-varying stress, called cyclical stress (Courtney 1990). The manufacturing of Ni–Ti endodontic instruments is more complex than that of stainless steel instruments, as the files have to be machined rather than twisted. This leads to surface irregularities (milling marks) and metal flash on the cutting edges that may potentially precipitate crack nucleation (Serene *et al.* 1995, Thompson 2000, Martins *et al.* 2002). Cracks were found to start at surface irregularities, scratches and inclusions which act as stress raisers during cyclic loading (Eggeler *et al.* 2004). As noticed by Kuhn *et al.* (2001), when the material is subjected to deformation or stress by machining, a high density of lattice defects, such as dislocations, is produced. Their X-ray diffraction and Vickers microhardness measurements on Ni–Ti instruments showed that the alloy is work-hardened.

It is important to mention that some authors employed metal guide tubes as artificial canals to simulate root canal geometry in studies of fatigue (Serene *et al.* 1995, Mize *et al.* 1998, Melo *et al.* 2002). Artificial canals prepared in this way may suffer wear and tear during testing and do not sufficiently restrict the instrument shaft, which will tend to regain its original straight shape, aligning itself into a trajectory of greater radius and less deformation. This may explain the higher average NCF values found in similar instruments after dry heat sterilization than those presented in this work. In the present work, analysis of the position of fracture in the instruments tested in fatigue, which occurred, on average, at 3 mm from the tip, without significant statistical difference between sterilized and nonsterilized instruments, indicates that the instruments always failed at the point of maximum tensile strain amplitude of the artificial canal, intentionally located at this point.

It was verified early on that the fatigue behaviour of Ni–Ti ProFile instruments is characteristic of low cycle fatigue (NCF $< 10^3$), where cracks are nucleated early in the process and that their slow propagation occupies the largest fraction of an instrument's fatigue life (Bahia & Buono 2005). Thus, it is possible that the observed increase in the NCF of the instruments subjected to sterilization is a result of a delay in fatigue crack nucleation and/or propagation, as the result of changes in the surface and subsurface defect structures generated during instrument machining. In fact, it is well known that deformation causes stabilization of martensite (Otsuka & Ren 2005). As no thermal treatment is applied to the instruments after machining, it is reasonable to expect that their surface and sub-surface regions contain stabilized martensite with a high concentration of dislocations. In the instruments fatigued to one-half of their fatigue life prior to sterilization, the amount of these defects is still higher. In both cases, the sterilization procedures may have caused dislocations annihilation and transformation of stabilized martensite back to austenite. This should occur in approximately the same degree for the two sterilization process employed, because the sterilization temperatures are well above the austenite finishing temperature Af, which is around 25 °C for nondeformed superelastic Ni–Ti (Bahia *et al.* 2005). Thus, the results obtained in this work are consistent with following reasoning: sterilization changed the surface and subsurface microstructure of the machined parts of the instruments, rendering these regions less susceptible to fatigue crack nucleation and propagation. No change in the bulk or on the nonmachined surfaces took place, as manifested by the lack of influence of sterilization on Vickers microhardness of the instruments and on tensile properties of the wires.

Conclusions

Changes in the mechanical properties of Ni–Ti endodontic instruments after five cycles of common sterilization procedures were insignificant. From the point of view of the clinician, the results obtained in this study suggest that these sterilization procedures are safe as they produced a significant increase in the fatigue resistance of new and used instruments.

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