In vitro resistance to fracture of two nickel-titanium rotary instruments made with different thermal treatments

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Summary

***Aim*. Aim of the study was to evaluate effectiveness of different heat treatments in improving Ni-Ti endodontic rotary instruments' resistance to fracture.**
***Methods*. 24 new NiTi instruments similar in length and shape: 12 M3 instruments, tip size 25 and .06 taper (United Dental, Shanghai, China), and 12 M3 Pro Gold instruments tip size 25 and .06 taper (United Dental, Shanghai, China), were tested in a 60° curved artificial root canal. Each group received a different heat treatment. Cycles to fracture were calculated for each instrument. Differences among groups were evaluated with an analysis of variance test (significance level was set at $P<0.05$.).**
***Results*. Statistical analysis found significant differences ($p<0.0213$) between groups. The M3 Pro Gold instruments were significantly more resistant to fatigue (mean values = 1012, SD +/- 77) than M3 instruments (mean values = 748, SD +/- 62). No statistically significant differences were found between fragments' lengths ($p>0,05$).**
***Conclusions*. An increased flexibility and the reduction of internal defects produced by heat treatments during or after manufacturing processes, may be responsible for improving resistance to cyclic fatigue and flexural stresses.**

Key words: endodontics, endodontic instruments, nickel-titanium, cyclic fatigue.

Introduction

In the last decades the introduction of nickel-titanium (NiTi) alloy in the manufacturing of endodontic instruments resulted in a significant improvement for root canal preparations which resulted easier in shaping procedures, faster, and more predictable (1-3). The superior mechanical properties of the NiTi alloy allowed the clinical use of NiTi rotary instruments with greater tapers (4, 5). This increase in dimensions and continuous rotation movements significantly improved effectiveness and rapidity of the cutting and simplified the achievement of a successful root canal treatment (6-8). Unfortunately, these features could also lead to an increased risk of intracanal separation of the instruments (9-11).

Several clinical and experimental studies demonstrated that multiple factors contribute to instrument's separation: cyclic fatigue has been proven to be one of the leading causes (12-14). Fatigue failure usually begins with the formation of microcracks that arise from the surface's irregularities of the instruments. During each loading cycle, microcracks develop and deepening until complete separation of the file (15-17). All NiTi rotary instruments show some irregularities and inner defects on the surface as a consequence of the manufacturing processes and the distribution of these defects influences their strength (18).

In recent years, manufacturers tried to find different solutions to develop instruments with enhanced resistance to flexural and torsional stresses, aiming at reducing the incidence of intracanal breakage. The three basic ways to achieve these improvements were mainly related to changes in design, heat treatments of the alloy and the use of reciprocating motions (19-23). Different and proprietary heat treatments have been developed and commercialized in the last decade, aiming at improving both flexibility and resistance to breakage, but they are not disclosed by manufacturers in detail (24). Changing the thermal history of the alloy, could produce an alloy with different characteristics for the endodontic use. By changing the thermal treatments, manufacturer can quickly and significantly modify clinical performance of NiTi instruments, with no need of modifications to the quality of the raw material or grinding machines. M3 Rotary and M3 Pro Gold are an example of similar instruments, produced by same manufac-

turer (United Dental, Shanghai, China), but with different performance due to different heat treatments.
The aim of the present study was to evaluate the hypothesis that different heat treatments can significantly affect the *in vitro* resistance to cyclic fatigue of NiTi rotary instruments. The null hypothesis was that no difference would be found between similar instruments with different heat treatments.

Material and methods

A total of 24 new NiTi instruments 25 mm in length was used in the present study: 12 M3 instruments, tip size 25 and .06 taper (United Dental, Shanghai, China) and 12 M3 Pro Gold instruments tip size 25 and .06 taper (United Dental, Shanghai, China). All instruments were the same in size and design, but they received a different heat treatment. All of them had been previously inspected using an optical stereomicroscope at x20 magnification for morphological analysis and checked for any signs of visible deformation. If defective instruments were found, they were discarded.
The cyclic fatigue testing device used in the present study has been used for previously performed studies on cyclic fatigue resistance (25, 26). The device consists of a mainframe to which is connected the electric handpiece and a stainless-steel block containing the artificial canal. The electric handpiece was mounted on a mobile device to allow precise and reproducible placement of each instrument inside the artificial canal to the same depth (18 mm) (Fig. 1). A simulated root canal with a 60° angle of curvature and 5 mm radius of curvature was used for all the tested instruments. All instruments were inserted at the same length (16 mm) and then rotated at 350 rpm with maximum torque until fracture occurred. For each instrument, the time to fracture was visually assessed and recorded with a 1/100 sec chronometer. Number of cycles to fracture was calculated for each instrument (NCF). Fragments were collected, measured and underwent to fractographic analysis performed by a scanning electron microscope (SEM) to determine fracture mode.
All data were recorded. For each group mean and standard deviations were calculated. Differences among groups were statistically evaluated with an analysis of variance test (significance level was set at $P<0.05$.). Data was statistically analyzed using the SPSS 17.0 software (SPSS Incorporated, Chicago, IL, USA).

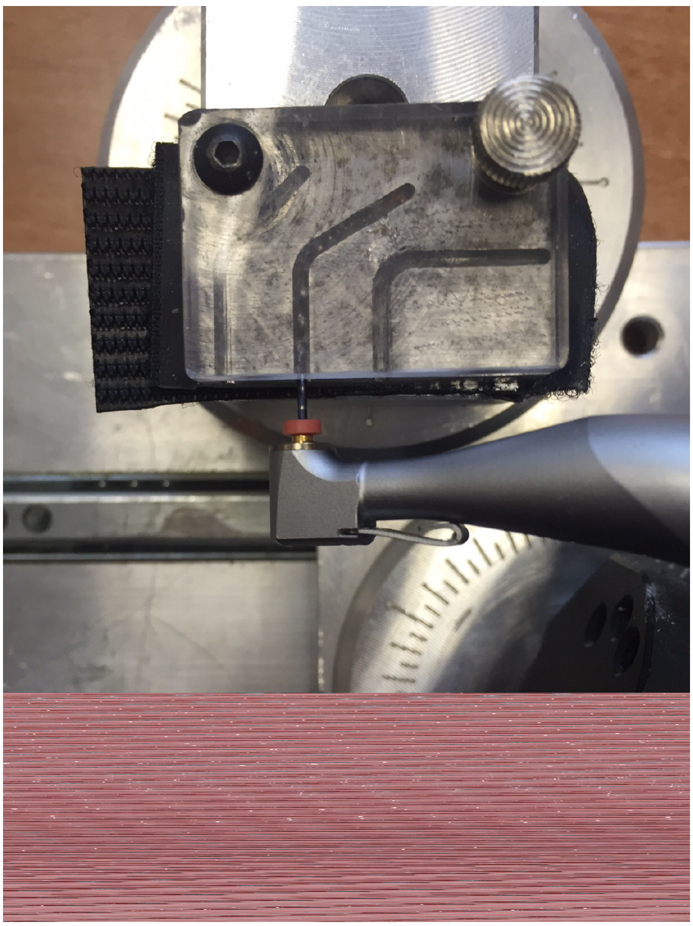

Figure 1. The testing device for cyclic fatigue.

Table 1. Results of cyclic fatigue tests (number of cycles to failure).

Group	NCF Mean Values	SD	Mean Fragment Lenght	SD
M3	748	(+/- 62)	5,4	(+/- 0,4)
M3 ProGold	1012	(+/- 77)	5,5	(+/- 0,5)

Table 2. Time to fracture in seconds (s).

Group	Seconds to fracture	SD
M3	128,3	(+/- 11,6)
M3 ProGold	173,5	(+/- 14,7)

Results

Results from the cyclic fatigue tests are shown in Tables 1 and 2. Mean values for time to fracture for M3 Pro Gold instruments were 173,5 seconds (SD +/- 14,7) and for M3 instruments were 128,3 seconds (SD +/- 11,6). Statistical analysis found significant differences ($p < 0.0213$) between the two groups. The M3 Pro Gold instruments were significantly more resistant to fatigue (NCF mean values = 1012, SD +/- 77) than M3 instruments (NCF mean values = 748, SD +/- 62). Mean value of the fragments' length for M3 Pro Gold instruments was 5,5 mm (SD +/- 0,5) and for M3 instruments was 5,4 mm (SD +/- 0,4). No statistically significant differences were found between fragment lengths ($p > 0,05$), showing proper insertion of the instruments inside artificial canals and consequently the same mechanical stresses applied on the same portions of the instruments (Fig. 2).

Discussion

NiTi endodontic alloys can exhibit three phases (27): the high-temperature B2 austenitic phase, the low-temperature B19' martensitic phase (monocyclic structure) and intermediate temperature R-phase (rhombohedral structure). Transformations of these phases are fundamental, because they determine the superelastic and shape memory characteristics of these instruments and their mechanical and functional properties and performance. These transformations can proceed by various ways, depending on the thermal history of the alloy during the manufacturing processes (18).
The heat treatment's parameters which are chosen to set the properties of the NiTi instruments are critical (23). In general, temperatures as low as 400°C and times as short as 1-2 minutes can set the shape, but generally, endodontic instrument's manufacturers prefer temperatures closer to 500°C for over 5 minutes. A rapid cooling of the instruments is preferred via water quench or rapid air cool. Longer heat treatment times and higher temperatures will increase the actuation temperature and often give a sharper thermal response (in the case of shape memory elements). However, there is usually a concurrent drop either in peak force (for shape memory alloys) or in

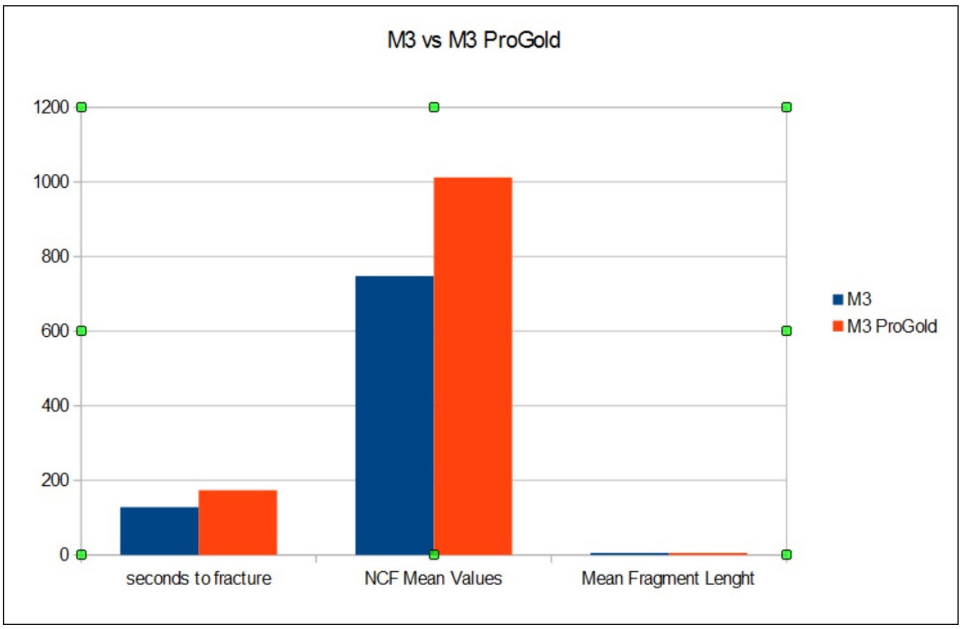

Figure 2. Differences between instruments' time to fracture, number of cycles to fracture (NCF) and lenght of the fractured segment

plateau stresses (for superelastic alloys). There is also an accompanying decrease in the ability of the instruments to resist permanent deformation (28).
Clinically, such changes result in different mechanical properties and behaviour. Even if the raw material and the machining processes are the same, the different and proprietary heat treatment exhibits more evident superelastic characteristics. On the contrary, M3 Pro Gold instruments are more ductile and softer, exhibiting more evident shape memory characteristics. Differently than M3, M3 Pro Gold can be easily precurved for easier insertion in curved canal and to reduce restoring forces. In the present study, the last characteristic allowed M3 Pro Gold instruments to better withstand the bending stresses. The null hypothesis was rejected: M3 Pro Gold instruments were found to be significantly more resistant to fatigue when compared to M3. The originality of the present study was in the possibility of testing instruments produced by the same manufacturer with the same NiTi alloy, but with different heat treatments. In the majority of previous studies about heat treated NTRIs, different instruments were tested from different manufacturers. In many cases, when manufacturers produce a new version of endodontic instruments with a different heat treatment, they also change instruments' design and this could make more difficult to evaluate the real effect of the heat treatment. The data are in accordance with previous studies which enlightened the importance of heat treatments in improving NiTi rotary instruments' resistance to breakage (12, 15, 18).
However, it is always difficult to compare different cyclic fatigue studies, because many factors can influence the final results. The main factor is the shape and dimension of the artificial canal (16). Each variation of canal curvature and diameter may affect the way that mechanical loads are applied and consequently different mechanical stresses on the instruments are induced (11). Moreover, also room temperature can dramatically influence fatigue resistance of the NTRIs, as shown in recent studies (29, 30). Therefore a correct comparison can only be made if the NTRIs are tested with the same device and artificial canal in the same conditions.
Fractographic analysis using SEM showed similar fracture patterns in both instruments. Metal fatigue commonly leads to ductile fracture with some plastic deformations and a typical dull dimpled surface (10). The dimpling involve the whole fracture surface, with some microvoids present as black dots (Figs. 3, 4). In ductile fracture, microvoids are produced inside the metal and their cohalescence weaken the structure and results in fracture.
NiTi instruments have constantly gained popularity during last decades because they offer more distinct clinical advantages with curved root canals than stainless-steel instruments, due to their higher flexibility, by virtue of their superelasticity. The superelasticity has made it possible to carry out conservative and better centered shapes, with less canal transportation and with more respect of the original anatomy (22). NiTi shape memory alloys undergo transformation from cubic austenitic to monocyclic martensite when the applied stress in the austenitic phase is enough to promote the stress-induced transformation (27). This stress-induced martensitic transformation reverses spontaneously upon release of the stress and the material returns to its original shape and size (23). Furthermore, the rhombohedral R-phase formation, thermoelastic phase, often precedes the martensitic transformation under certain conditions and is considered to be the main reason for increased flexibility of NiTi instruments over traditional stainless steel instruments. Such an increased flexibility and the reduction of internal defects produced by heat treatments during or after manufacturing processes, are responsible for greater resistance to flexural

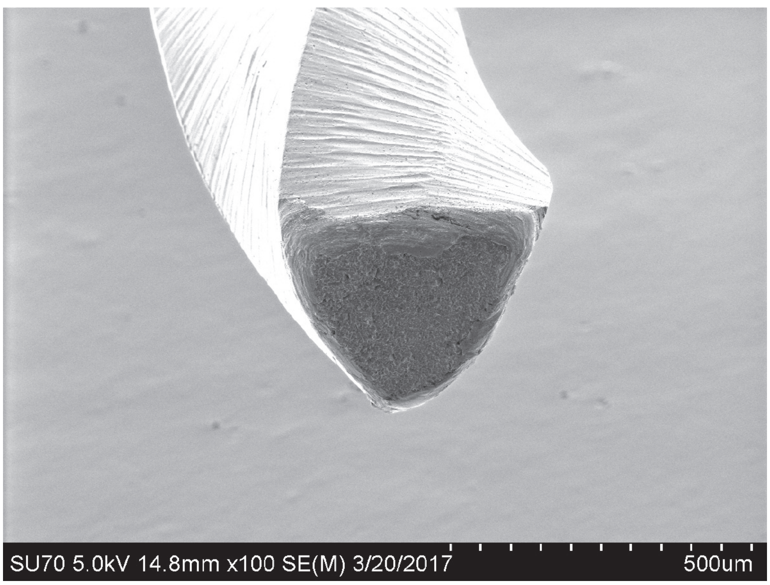

Figure 3. SEM microscope image of a fractured M3 Pro Gold instrument.

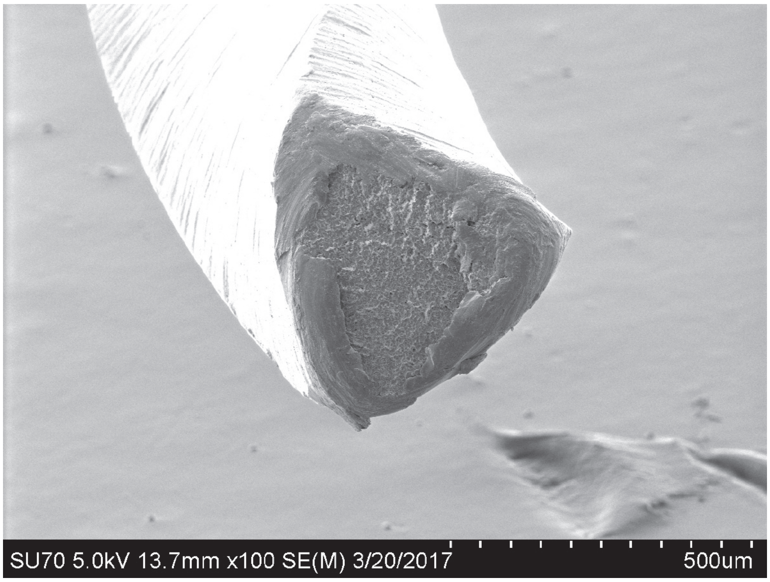

Figure 4. SEM microscope image of a fractured M3 Pro Gold instrument.

stresses and consequently to cyclic fatigue. Further studies, however, are needed to evaluate how much different heat treatments affect torsional resistance and hardness of the alloy in NiTi rotary instruments.

Conclusions

Changing the thermal treatments, allows manufacturers to quickly and significantly improve clinical performance of NTRIs, with no need of changing quality of the raw material or modifying the grinding machines. M3 Rotary and M3 Pro Gold are an example of NTRIs with same design and alloy, produced by the same manufacturer, which show different resistance to cyclic fatigue due to different heat treatments.

Declaration of conflicting interests

The Authors declare that there is no conflict of interest.

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