Influence of heat treatment on fatigue resistance of two NiTi endodontic files

Influenza del trattamento termico sulla fatica di due strumenti endodontici in NiTi

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ABSTRACT

OBJECTIVES

To evaluate the cyclic, torsional fatigue resistance and phase transformation of two heat-treated and non-heat-treated nickel-titanium reciprocating instruments.

MATERIALS AND METHODS

Twenty non-heat-treated (Procodile, Komet, Brasseler GmbH & Co., Lemgo, Germany) and 20 heat-treated (Procodile Q, Komet, Brasseler GmbH & Co., Lemgo, Germany) files (25 mm length, #25 apical diameter and 0.6 taper) were subjected to fatigue resistance tests.

The dynamic cyclic fatigue was tested at 35 ± 1 °C, using a dedi-

cated patented device, in an artificial stainless-steel canal with a 60° angle of curvature, the plate containing the artificial canal performing a controlled axial upand-down movement at 8 mm/s speed.

The instruments were operated with a specific reciprocating motion (Reflex Dynamic[®], Komet, Breasseler GmbH & Co., Lemgo, Germany). Time to fracture (TtF) was recorded and the length of the fractured tips was measured. The torsional fatigue resistance was tested at room temperature (21±1 °C) using a custom-made device manufactured according to ISO 3630-1. The instruments were fixed 3 mm from the tip and their shafts were rotated counterclockwise at a speed of 2 rpm until fracture. The maximum torque load (Ncm) and corresponding rotation angle at fracture were recorded.

All the results were statistically analysed (p < 0.05). Fractographic analysis was performed using a field emission gun scanning electron microscope (FEG-SEM) to disclose the fractured surface characteristics.

To assess the temperature range for phase transformations, differential scanning calorimetry (DSC) was performed on small segments of \approx 20 mg mass subjected to 2 heating and 2 cooling thermal cycles at rates of 5 °C min⁻¹ in flowing N_2 atmosphere over a temperature range of -40 °C to +110 °C.

RESULTS

The heat-treated instruments showed a greater resistance to dynamic cyclic fatigue as compared to the non-heat-treated sample (TtF 303 ± 18.5 s *vs* 220 ± 18.4 s; p <0.05) and a higher resistance to torsional fracture, bearing a greater maximum torque load (1.67 ± 0.16 *vs* 0.82 ± 0.07 Ncm; p <0.05). No significant differences were detected between heat-treated and

non-heat-treated samples in mean angular rotation to fracture ($298\pm25^{\circ}$ vs $312\pm32^{\circ}$; p >0.05) and in the mean length of the fractured fragments (p >0.05). All instruments showed both ductile and brittle fracture patterns.

According to the DSC, the direct (cooling) and reverse (heating) transformations of the non-heattreated files occurred at lower temperatures (<25 °C) than those

RIASSUNTO

OBIETTIVI

Valutare la resistenza alla fatica ciclica e torsionale e la trasformazione di fase di due strumenti endodontici reciprocanti in nichel-titanio, trattati e non trattati termicamente.

MATERIALI E METODI

Venti strumenti endodontici non trattati termicamente (Procodile, Komet, Brasseler GmbH & Co., Lemgo, Germania) e 20 strumenti trattati termicamente (Procodile Q, Komet, Brasseler GmbH & Co., Lemgo, Germania), di lunghezza 25 mm, diametro apicale #25 e conicità 0.6, sono stati sottoposti a prove di resistenza alla fatica. La fatica ciclica dinamica è stata testata con un dispositivo sperimentale brevettato, a una temperatura di 35±1 °C, in un canale artificiale in acciaio inossidabile con curvatura di 60°, che eseguiva un movimento assiale controllato, a una velocità di 8 mm/s. Gli strumenti sono stati utilizzati con uno specifico movimento reciprocante (Reflex Dynamic[®], Komet, Breasseler GmbH & Co., Lemgo,

of the heat-treated files (≈50 °C), the latter thus not being austenitic at room and body temperatures at which they are operated. Also, the different transformation

enthalpies suggest a multi-step transformation, likely involving R-phase formation, for heat-treated files, against a direct transition between austenitic and martensitic phase for non-heat-treated files.

Germania). Sono stati misurati il

tempo di frattura (TtF) e le lun-

CONCLUSIONS

According to these findings, heat treatment of the tested files provides them with microstructural properties more suited to the clinical operating conditions and improved performances in terms of torsional and flexural strength.

CLINICAL SIGNIFICANCE

Heat-treated files might be the best choice, over the traditional

non-heat-treated files, when facing challenging clinical conditions, such as curved and constricted canals.

KEY WORDS

- Cyclic fatigue resistance
- Heat-treatment
- NiTi endodontic instruments
- Reciprocating
- Torsional resistance

ghezze dei segmenti apicali fratturati. La resistenza alla fatica torsionale è stata testata a temperatura ambiente (21±1 °C), vincolando gli strumenti a 3 mm di distanza dall'estremità e applicando loro una rotazione in senso antiorario a una velocità di 2 rpm, fino alla frattura. Sono stati registrati il carico di coppia massimo n- (Ncm) e l'angolo di rotazione corrispondente alla frattura. Tutti i risultati sono stati analizzati stati-

(Ncm) e l'angolo di rotazione corrispondente alla frattura. Tutti i risultati sono stati analizzati statisticamente (p < 0,05). L'analisi frattografica è stata eseguita con un microscopio elettronico a scansione con sorgente a emissione di campo (FEG-SEM). Per studiare le temperature di trasformazione di fase è stata eseguita la calorimetria differenziale a scansione (DSC), in un intervallo da -40 °C a +110 °C, alla veloci-

RISULTATI

tà di 5 °C min-1.

Gli strumenti trattati termicamente hanno mostrato una maggiore resistenza alla fatica ciclica dinami-

ca rispetto agli strumenti non trattati termicamente (TtF 303±18,5 s vs 220±18,4 s; p <0.05) e una maggiore resistenza alla frattura torsionale, sopportando un carico di coppia massimo maggiore (1,67±0,16 vs 0,82±0,07 Ncm; p <0,05). Non sono state rilevate differenze significative nell'angolo di rotazione alla frattura tra i campioni trattati termicamente e quelli non trattati termicamente (298±25° vs 312±32°; p >0,05), né nella lunghezza media dei frammenti fratturati (p >0,05). Tutti gli strumenti hanno mostrato pattern di frattura sia di tipo duttile che fragile.

Secondo la DSC le trasformazioni dirette e inverse degli strumenti non trattati termicamente sono avvenute a temperature inferiori (<25 °C) rispetto a quelle degli strumenti trattati termicamente (≈50 °C). È verosimile, quindi, che questi ultimi non siano in fase austenitica alle temperature ambiente e corporea alle quali vengono utilizzati. Inoltre, la differenza nei valori di entalpia suggerisce una trasformazione a più stadi per gli strumenti trattati termicamente, con formazione intermedia della fase R, rispetto a una transizione diretta tra fase austenitica e martensitica per gli strumenti non trattati termicamente.

CONCLUSIONI

Secondo i risultati di questo studio il trattamento termico degli strumenti testati conferisce loro proprietà microstrutturali più adatte alle condizioni operative cliniche e migliori prestazioni in termini di resistenza alla torsione e alla flessione.

SIGNIFICATO CLINICO

Gli strumenti trattati termicamente potrebbero essere la scelta migliore, rispetto a quelli tradizionali non trattati termicamente, quando si devono affrontare condizioni cliniche complesse, come il trattamento di canali curvi e stretti.

PAROLE CHIAVE

- Fatica ciclica
- Trattamento termico
- Strumenti endodontici in NiTi
- Strumenti reciprocanti
- Fatica torsionale

1. INTRODUCTION

Compared to stainless steel files, nickeltitanium (NiTi) endodontic instruments have numerous advantages, such as greater flexibility and the capacity to follow the root canals without making ledges or perforations^[1]. Despite these advantages, NiTi instruments can unexpectedly fracture during the root canal shaping, mainly for flexural or torsional fatique^[2,3]. The fracture of NiTi files inside the root canal may suddenly happen without any noticeable warning^[4]. This negatively influences the prognosis of the endodontic therapy, especially if a periapical radiolucency is present^[5]. The torsional fracture occurs when the file tip is locked inside the root canal and the shank continues to rotate^[6], or when the torque resulting from the contact between the instruments and the canal wall exceeds the elastic limit of the alloy^[2]. Differently, the flexural or cyclic fatigue fracture is determined by continuous cycles of compressive and tensile stresses on a file which rotates inside the curved canal without bindings^[7].

Many factors can affect the fatigue resistance of NiTi rotary files, i.e. the size and taper of the file, the radius and angle of curvature of the root canal^[8], the alloys, the kinematics and the thermal treatment. The reciprocation motion, consisting of an alternate oscillation of the file in both rotation directions, may increase the cyclic fatigue resistance of the file^[9], compared to the omnidirectional rotation. This type of motion reduces the risk of fracture, as well as the operating time, even for inexperienced operators^[2,10]. In addition to the kinematics. the thermal treatment of the files has already been shown to improve the flexibility, cutting efficiency and canal centering ability^[11].

Therefore the aim of this study was to evaluate the influence of heat treatment on cyclic and torsional fatigue resistance of two nickel titanium reciprocating instruments and to correlate the results with their transformation temperature.

2. MATERIALS AND METHODS Sample size calculation

The sample size estimate was calculated a priori using G*Power 3.1.9.6 software (Heinrich-Heine-Universität Düsseldorf, Germany). Considering a test power of 0.80 with a = 0.05 and effect size = 0.85, twenty instruments of each type (n = 20) were submitted to a dynamic cyclic fatigue test at 35 ± 1 °C and a torsional resistance test at 21 ± 1 °C.

Three segments of each instrument per type, between 15 and 20 mg of weight, were used for differential scanning calorimetry (DSC).

Dynamic cyclic fatigue test

For the purposes of this study, 20 nonheat-treated-group 1 (Procodile, Komet, Brasseler GmbH & Co., Lemgo, Germany) and 20 heat-treated shaping filesgroup 2 (Procodile Q, Komet, Brasseler GmbH & Co., Lemgo, Germany) were tested for their dynamic cyclic fatigue resistance. All the tested instruments have the same design and features (25 mm length, #25 apical diameter and 0.6 taper), and only differ for the heat-treatment. All instruments were examined under a stereomicroscope (Leica EZ-4D, Wetzlar, Germany) prior to the experiment to detect any defects or deformations and none of the instruments were discarded

Each instrument was rotated until fracture inside an artificial canal, using a custom-made device specifically designed and assembled to test dynamic cyclic fatigue at controlled temperature (University of Modena and Reggio Emilia, patent no. 10202000008560).

The artificial canal has the same geometry and taper as the tested endodontic instruments, with an increased diameter of approximately 150 µm and a 5 mm long curvature with a 5 mm radius and a 60° angle: the curvature center is 5 mm from the instrument tip. The stainlesssteel plate containing the artificial canal features an axial up-and-down movement at 8 mm/s speed, simulating the instrument excursion during the clinical procedure. The axial movement of the plate is controlled by means of a linear actuator, the position of which is dynamically controlled via a potentiometer. Both the experimental plate containing the artificial canal and the instrument being tested are immersed in osmotic water obtained from a thermostatic tank at 35±1 °C. An electric pump ensures a continuous water exchange (1200 l/h) in the tank, allowing the temperature to be maintained at a constant level. Moreover, the temperature is constantly monitored by a thermic probe for the entire duration of the test; the thermic probe is used for controlling the thermostatic bath as well. Whenever temperature is lower than the given threshold, a waterproof resistor in the thermostatic tank is switched on; the system is controlled by means of a Schmitt trigger implemented in a Matlab® (MathWorks Inc., Natick, MA, USA) code.

The endodontic motor handpiece is fixed to a holder specifically designed to ensure the reproducibility of the handpiece positioning and of the inclination of the endodontic instrument in relation to the artificial canal. For the experimental procedure the tested instruments were inserted in the artificial canal in a standard position (0°) (fig. 1).

The endodontic motor EndoPilot (Komet, Brasseler GmbH & Co., Lemgo, Germany) was used to operate the instruments with the specifically designed Reflex Dynamic[®] (Komet, Brasseler GmbH & Co., Lemgo, Germany) reciprocating motion. This kinetic consists of a continuous counterclockwise rotation, interrupted by pauses of 30 milliseconds, alternated with clockwise movements, occurring whenever the software detects excessive resistance into the canal. All instruments were rotated until fracture.

All the start-up and control stages of the test are managed by a dedicated software developed within Matlab® framework, and all the tests are recorded by a camera, which allows for the time to fracture (TtF) to be calculated, avoiding human errors. The same software is able to collect all data related to the test (such as test duration, temperature, linear velocity and travel distance of the actuator) and to store them for further analyses. The goal of this approach is to ensure a better repeatability of the fatigue tests. The length of the fractured fragment was measured by digital imaging with a Leica EZ-4D stereomicroscope (Leica, Wetzlar, Germany) and Fiji software (National Institutes of Health, Bethesda, MD, US) to further verify the correct positioning of the instrument in the artificial canal.

The Shapiro-Wilk test was performed to verify the normal distribution of data, then the Student's T test was used to compare the two groups. The Stata 11 (StataCorp, College Station, TX, USA) software was employed for these analyses, p value was set at 0.5.

Torsional fatigue test

Another sample of 25 mm long non-heat-treated (group 1, n = 20) Procodile (Komet, Brasseler GmbH & Co., Lemgo, Germany) and heat-treated (group 2, n =20) Procodile Q (Komet, Brasseler GmbH & Co., Lemgo, Germany) endodontic instruments (size 25# and 0.6 ta-



Fig. 1 The cyclic fatigue test device specifically designed for the dynamic cyclic fatigue test at 35 ± 1 °C. The detail in the lower right side shows the artificial canal designed to test the non-heat-treated and heat-treated 25, .06 files

per) were tested for their torsional fatigue resistance.

Each instrument was inspected for defects or deformities prior to the experiment using a stereomicroscope (SZR-10, Optika, Bergamo, Italy) and no instrument was discarded. A torsional load was applied until fracture to evaluate the average final torsional strength and angle of rotation of the instruments, using a custom-made device manufactured according to ISO 3630-1 as already described in previous published studies^[12].

Each instrument was fixed 3 mm from the tip using a spindle connected to a torque-sensitive load cell; the shaft of the instrument was then fixed in an opposing spindle designed to be rotated with a stepper motor. The shaft of Procodile and Procodile Q was rotated counterclockwise at a speed of 2 rpm until the instrument separation occurred. The peak torque load (Ncm) and the corresponding angular rotation (°) were continuously monitored using a tensiometer (Sabri Dental Enterprises, Downers Grove, IL) at room temperature (21±1 °C) and the maximum torsional fracture resistance and angle of rotation were recorded.

First, the normality of the distribution and the homogeneity of the variances of the data were checked using the Kolmogorov-Smirnov's and Levene's tests, respectively. The data were then statistically analysed using analysis of variance tests and the Student-Newman-Keuls test for multiple comparisons (Prism 5.0, GraphPad Software, Inc., La Jolla, CA, USA) with a significance level set at 5% (p < 0.05).

Fractographic analysis

Instruments submitted to cyclic and torsional fatigue tests were cleaned by immersion in an ultrasonic bath containing absolute alcohol for 5 minutes to remove possible debris. The instruments were observed using a field emission gun scanning electron microscope (FEG-SEM: Nova NanoSEM 450, FEI Company-Oxford Instruments, Eindhoven, NL) at 600x and 5000x to reveal failure mechanisms.

Differential scanning calorimetry

To assess the temperature range for phase transformations, differential scanning calorimetry (DSC-Q2000, TA Instrument, New Castle, DE, USA) was performed. The working parts of new non-heat-treated Procodile and heattreated Procodile Q instruments were wire-cut into small segments and samples with a mass of ~20 mg were placed in an aluminum crucible, with an empty crucible as a reference. The sample and reference were both subjected to 2 heating and 2 cooling thermal cycles at heating/cooling rates of 5 °C min-1 in flowing N₂ atmosphere over a temperature range between -40 °C and +110 °C. Thermal plots were analyzed using the Universal Analysis 2000 (TA Instrument, New Castle, DE, USA) software to obtain the onset temperatures of phase transformations and the associated enthalpy changes (ΔH). Four transformation temperatures were recorded for each file: martensite start (M_{s)}, martensite finish (M,), austinite start (A,, and austinite finish (A,).

3. RESULTS

Dynamic cyclic fatigue test

The mean TtF values measured in seconds were 220 ± 18.4 s for non-heat-

treated 25.06 files (group 1) and 303 ± 18.5 s for the heat-treated 25.06 files (group 2) (tab. I).

Heat-treated files (group 2) showed a si-



Figg. 2a-d Field-emission gun scanning electron microscopy (FEG-SEM) micrographs analysis showing the fractured specimens (axial views) of (a, c) non-heat-treated files (group 1), size 25, .06 taper and (b, d) heat-treated files (group 2), size 25, .06 taper after cyclic fatigue testing at 35 ± 1 °C (a, b) and after torsional testing at room temperature (c, d). Dotted lines (a, b) highlight the brittle crack propagation area while circles (c, d) indicate concentric abrasion marks, typical features of torsional failures. Round insets show high magnification details of the dimples area

Tab. I Mean±standard deviation of the time to fracture (TtF 35±1 °C), torque (Ncm) and angle of rotation (°) of the tested instruments

Instrument	TtF (s)	Torque (N·cm)	Angle of rotation (°)	
Group 1 (non-heat-treated)	303±18.5ª	0.82±0.07ª	312.91±32ª	
Group 2 (heat-treated)	220±18.4 ^b	1.67±0.16 ^b	298.66±25ª	

Different superscript letters in the same column indicate statistically significant differences among groups (p <0.05)

gnificantly greater resistance to cyclic fatigue (p < 0.05) as compared to non-heattreated files (group 1).

There was no significant difference in the mean length of the fractured fragments for all the instruments tested (p >0.05). The fracture morphologies (**figg. 2a-d**) allowed to identify the crack initiation/ propagation areas and the final fracture zone. All instruments showed both ductile and brittle fractured surface.

Torsional fatigue test

The mean values and standard deviations of the maximum torque load (Ncm) were 0.82 ± 0.07 Ncm and 1.67 ± 0.16 Ncm for non-heat-treated (group 1) and heat-treated instruments (group 2), respectively. The corresponding rotation angle up to fracture was similar for both the tested instruments, yielding a mean value and standard deviation of 312.91±32° for the nonheat-treated (group 1) and 298.66±25° for the heat-treated group (group 2).

Heat-treated files (group 2) showed a higher resistance to torsional fracture (p <0.05), bearing a greater maximum load until fracture. No significant differences were detected between the two groups when comparing the values of angular rotation leading to fracture (p >0.05). Similar fracture patterns were observed in the two groups, with both brittle and ductile components (**figg. 2a-d**). ting) transformations of non-heat-treated instruments (group 1) occurred at lower temperatures than those of heat-treated ones (group 2) and both the A_f and the M_s temperatures for the non-heat-treated files were below 25 °C (tab. II).

 A_{r} and M_{s} temperatures for heat-treated instruments, by contrast, were much higher, at around 50 °C (**tab. II**), with the direct transformation ending at ≈ 20 °C (M_{r} temperature) and the reverse one beginning at ≈ 26 °C (A_{s}).

Differential scanning calorimetry

DSC scans showed significant differences between the two file types (**figg. 3a**, **b**). The direct (cooling) and reverse (hea-

4. DISCUSSION

Changes in the designs, kinematics and manufacturing procedures such as heat treatments have shown to improve the mechanical properties of the NiTi alloys of endodontic instruments^[8,11]. Particu-



Figg. 3a, b DSC curves of (a) non-heat-treated-group 1 and (b) heat-treated-group 2 instruments. Heat flow per mass unit of sample (ordinate) *versus* temperature (abscissa) for direct transformation (cooling) and inverse (heating). The overall variation of entropy per mass unit associated with each transformation (dH) is indicated on the graphs

Tab. II Average phase transformation temperature and enthalpy changes (Δ H) of the tested instruments									
Instrument	Cooling			Heating					
	M _s (°C)	M _f (°C)	ΔH (J/g)	А _s (°С)	Α _f (°C)	∆H (J/g)			
Group 1 (non-heat-treated)	22.64	-2.83	2.6	4.60	23.70	2.7			
Group 2 (heat-treated)	49.33	19.91	3.6	26.13	50.09	3.3			

larly, characteristics such as cyclic fatigue resistance and torsional fatigue resistance have a major impact on the clinical performances of endodontic files^[2,5]. In this study we evaluated such properties, along with the analysis of fracture morphology and a DSC characterization, on two file systems with the same geometrical and operational features, only differing for the heat treatment they underwent during the manufacturing process, thus allowing a direct comparison between the two and the isolation of the heat treatment variable.

According to the DSC thermograph, the non-heat-treated files (group 1) tested are expected to be fully austenitic at room temperature and even more at the human body temperature. As a side note, the transformation temperatures of such files detected in this study were slightly higher than those we had measured in a previous study using the same experimental setting^[13]. This might imply that slight changes have meanwhile occurred to the manufacturing process of the base NiTi wire used for these files. As a matter of fact, the non-heat-treated files tested in that study^[13] featured a green-coloured coating, based on a niobium/niobium oxide bilayer, which is instead absent from the present samples, and this might further corroborate the idea that some process change has meanwhile been introduced.

The higher A_r and M_s temperatures showed by the tested heat-treated instruments (group 2) indicate that such files are certainly not in the austenite phase both at room temperature and at the human body temperature at which they are operated. Moreover, such files exhibited significantly higher transformation enthalpies (ΔH , **tab. II**) than the non-heattreated files (group 1). This suggests that the austenite-martensite transformation paths for the two files were different. Specifically, the higher transformation enthalpy of heat-treated instruments (group 2) suggests a multi-step transformation, likely involving the formation of the R-phase, although the two transformation ranges overlapped to the point that they were not clearly distinguishable in the DSC scans (**fig. 3b**).

Non-heat-treated files (group 1) should, instead, exhibit a direct transition between the two phases. It is therefore inferred that the heat-treated instruments (group 2) should be mainly in the R-phase at human body temperature, and they might still contain substantial amounts of Rphase together with martensite at room temperature.

While many studies have assessed the static cyclic fatigue resistance^[12,14], in this study we used a new custom-made patented system to evaluate dynamic cyclic fatigue instead. This choice, in addition to using a testing system maintained at a constant temperature of 35 ± 1 °C, allows for an optimal and realistic reproduction of the clinical setting during the procedure. Also, the provided "pecking" movement, results in a more even and realistic stress distribution along the entire length of the tested instrument^[15].

While other authors tested more extreme conditions such as 90° canal curvature^[15,16], in this study the morphology of the canal (60° curvature) was chosen to reproduce one of the unfavourable anatomical conditions that can be encountered in clinical practice at which fracture can occur, as described by Pruett et al.^[16,17].

The contribution of kinematics to cyclic fatigue resistance is also to be mentioned: both the instruments we tested were operated with a specifically designed reciprocating movement, which combines the advantages of rotary and reciprocating motion^[18].

Reciprocating kinematics, regardless of the specific configuration of reciprocating motion, have shown significantly higher flexural resistance than traditional rotating movements^[9]. As a matter of that, both the tested instruments showed TtF values similar to other reciprocating instruments^[16].

The better performances of the heat-treated files (group 2), as compared to nonheat-treated ones (group 1), in terms of dynamic cyclic fatigue are thus attributable to the specific heat treatment. According to DSC findings, heat-treated instruments (group 2) are expected to be mostly in their martensitic or R-phase at clinical operating temperatures, suggesting that they exhibit greater flexural resistance, thus possibly achieving better clinical performances^[19].

Similarly, Topçuoğlu et al. using the same setting, found similar outcomes when comparing heat-treated *versus* non-heat-treated files^[20].

The mean length of the fractured portion was not significantly different between the two tested files, confirming that the instruments were correctly positioned in the experimental device^[21].

As for torsional fatigue, no significant differences in the angular rotation between the two instruments were found, while the torsional resistance was significantly higher for the heat-treated group. These findings might be explained by the fact that the two instruments are made of the same alloy and have the same geometrical features, thus leading to the same behaviour in terms of angular rotation to fracture, but they only differ for the heat treatment, which likely confers a higher failure torque. Conversely, a previous study^[12] comparing two reciprocating files of the same manufacturer, only differing for the heat treatment of one of them, found higher torsional resistance for the nonheat-treated files. These discrepancies might however be due to the specific heat treatment.

According to fractographical analysis, all instruments similarly exhibited two well

distinguishable areas with different morphologies, indicating two main fracture mechanism. The area showing sharp edged grains indicates a brittle-type propagation of the fracture, while the one displaying plastically deformed dimples indicates a more ductile behaviour. These findings suggest that through an early brittle propagation, cracks propagate in the material reducing the resistant section, until the applied load leads to a final ductile separation of the parts^[22]. Furthermore, it appears that the heat treatment did not have a significant impact on the fracture propagation behaviour in the two instruments.

5. CONCLUSIONS

Within the limits of the present experimental setting, the results of this study indicate that the heat treatment of the tested files (group 2) provides them with microstructural properties more suited to the clinical operating conditions and improved performances in terms of torsional and flexural strength.

CONFLICT OF INTEREST

The authors confirm that are no known conflicts of interest associated with this publication.

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REFERENCES

1. Keskin C, Inan U, Demiral M, Keleş A. Cyclic fatigue resistance of Reciproc Blue, Reciproc, and WaveOne Gold reciprocating instruments. J Endod 2017 Aug;43(8):1360-3.

2. Generali L, Righi E, Todesca MV, Consolo U. Canal shaping with WaveOne reciprocating files: influence of operator experience on instrument breakage and canal preparation time. Odontology 2014 Jul;102(2):217-22.

3. Iacono F, Pirani C, Generali L, Sassatelli P, Nucci C et al. Wear analysis and cyclic fatigue resistance of electro discharge machined Ni-Ti rotary instruments. G Ital Endod 2016 Jun;30(1):64-8.

4. Zhou H Min, Shen Y, Zheng W, Li L, Zheng Y Feng, Haapasalo M. Mechanical properties of controlled memory and superelastic nickel-titanium wires used in the manufacture of rotary endodontic instruments. J Endod 2012 Nov;38(11):1535-40.

5. Panitvisai P, Parunnit P, Sathorn C, Messer HH. Impact of a retained instrument on treatment outcome: a systematic review and metaanalysis. J Endod 2010 May;36(5):775-80.

6. Peters O. Current challenges and concepts in the preparation of root canal systems: a review. J Endod 2004 Aug;30(8):559-67.

7. Ounsi HF, Salameh Z, Al-Shalan T, Ferrari M, Grandini S et al. Effect of clinical use on the cyclic fatigue resistance of ProTaper nickeltitanium rotary instruments. J Endod 2007 Jun;33(6):737-41. 8. Higuera O, Plotino G, Tocci L, Carrillo G, Gambarini G, Jaramillo DE. Cyclic fatigue resistance of 3 different nickel-titanium reciprocating instruments in artificial canals. J Endod 2015 Jun;41(6):913-5.

9. Pedullà E, Grande NM, Plotino G, Gambarini G, Rapisarda E. Influence of continuous or reciprocating motion on cyclic fatigue resistance of 4 different nickel-titanium rotary instruments. J Endod 2013 Feb;39(2):258-61.

10. van der Vyver PJ, Jonker C. Reciprocating instruments in endodontics: a review of the literature. SADJ 2014 Oct;69(9):404-9.

11. Pereira ESJ, Gomes RO, Leroy AMF, Singh R, Peters OA et al. Mechanical behavior of M-Wire and conventional NiTi wire used to manufacture rotary endodontic instruments. Dent Mater 2013 Dec;29(12):e318-24.

12. Generali L, Puddu P, Borghi A, Brancolini S, Lusvarghi L, Bolelli G et al. Mechanical properties and metallurgical features of new and *ex vivo* used Reciproc Blue and Reciproc. Int Endod J 2020 Feb;53(2):250-64.

13. Generali L, Malovo A, Bolelli G, Borghi A, La Rosa GRM et al. Mechanical properties and metallurgical features of new green NiTi reciprocating instruments. Materials 2020 Aug 24;13(17):3736.

14. Pedullà E, Corsentino G, Ambu E, Rovai F, Campedelli F et al. Influence of continuous rotation or reciprocation of optimum torque reverse motion on cyclic fatigue resistance of nickel-titanium rotary instruments. Int Endod J 2018 May;51(5):522-8.

15. Elsewify T, Elhalabi H, Eid B. Dynamic cyclic fatigue and differential scanning calorimetry analysis of R-Motion. Int Dent J 2023 Jan;S0020-6539(22)00284-2.

16. Hülsmann M, Donnermeyer D, Schäfer E. A critical appraisal of studies on cyclic fatigue resistance of engine-driven endodontic instruments. Int Endod J 2019 Oct;52(10):1427-45.

17. Pruett JP, Clement DJ, Carnes DL. Cyclic fatigue testing of nickel-titanium endodontic instruments. J Endod 1997 Feb;23(2):77-85.

18. Mena-Álvarez J, Almanzor-López M, Quispe-López N, De Pedro-Muñoz A, Rico-Romano C. Analysis of the importance of the motion used in the resistance of different mechanical instrumentation systems in endodontics: a comparative study. Materials 2022 Jun 24;15(13):4443.

19. Zupanc J, Vahdat-Pajouh N, Schäfer E. New thermomechanically treated NiTi alloys - a review. Int Endod J 2018 Oct;51(10):1088-103.

Topçuoğlu HS, Düzgün S, Aktı A, Topçuoğlu G. Laboratory comparison of cyclic fatigue resistance of WaveOne Gold, Reciproc and WaveOne files in canals with a double curvature. Int Endod J 2017 Jul;50(7):713-7.
Plotino G, Grande NM, Testarelli L, Gambarini G, Castagnola R, Rossetti A. Cyclic fatigue of Reciproc and Reciproc Blue nickel-titanium reciprocating files at different environmental temperatures. J Endod 2018 Oct;44(10):1549-52.

22. Cheung GSP, Peng B, Bian Z, Shen Y, Darvell BW. Defects in ProTaper S1 instruments after clinical use: fractographic examination. Int Endod J 2005 Nov;38(11):802-9.