

Original Research

Influence of short-term cooling on the performance of superelastic and thermally-treated rotary NiTi files tested in dynamic cyclic fatigue model

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1. Abstract

Aim: The purpose of the present study was to investigate the effects of various short-term cooling durations on the performance of both superelastic and thermally treated nickel titanium (NiTi) files, tested in a dynamic cyclic fatigue model. **Methodology:** Superelastic RaCe (FKG, La Chaux-de-Fonds, Switzerland) and thermally treated RaCe EVO (FKG, La Chaux-de-Fonds, Switzerland) files of the same size and taper (25, 0.06) (n = 45 each) were tested using a dynamic cyclic fatigue model, where the number of cycles to failure (Nf) was measured at simulated body temperature. In each group, the samples were tested as received, after 5 seconds of cooling treatment, and after 15 seconds of cooling treatment (n = 15 each). Fractured file surfaces were investigated via scanning electron microscopy. Statistical analysis was performed using a one-way ANOVA with Scheffe's post hoc test at a significance level of 0.05. **Results:** Significant differences in the Nf between the tested groups were found ($p < 0.05$) at each testing condition, where RaCe Evo files showed overall improved cyclic fatigue resistance. Only the RaCe groups at 5 second cooling showed significantly higher Nf than the control subgroup ($p < 0.05$). RaCe EVO group, showed no significant difference between the 3 subgroups ($p > 0.05$).

Scanning Electron Microscope (SEM) images revealed typical features of cyclic fatigue behavior in both groups. **Conclusions:** Short-term cooling application for 5 seconds on superelastic NiTi files showed an enhanced cyclic fatigue resistance.

2. Introduction

Due to their superior properties, nickel titanium files have become a prominent part of the endodontic arsenal to facilitate the instrumentation of root canals [1]. The advent of NiTi endodontic files has made it possible to attain preparations that are larger at the apical part, more centered, and with reduced incidence of deviations compared to stainless-steel files [2, 3]. Despite their advantages, NiTi rotary files may undergo premature failure leading to fracturing [4].

There are two essential causes of instrument separation in the canal, excessive torsional and/or flexural load that generate stresses that exceed the elastic deformation capacity of the instrument, leading it firstly to a plastic deformation and secondly to fracture. Torsional fatigue occurs when the tip of the instrument binds in the root canal while the shank continues to rotate [2]. These two stresses, on the

other hand, are better considered together, and a complete analysis of the behavior of rotating instruments is possible considering the polar moment of inertia [5].

The mechanical behavior of NiTi alloys is related to the proportions and the characteristics of the main microstructural phases, which are austenite, martensite, and R phases. The fatigue resistance of NiTi endodontic files is affected by the transition temperatures of the NiTi alloys that are mainly adjusted with heat treatment [6]. The mechanical performance is also dependent on the surrounding temperature. If the temperature is above austenite finish temperature (A_f), the alloy will be in an austenitic state which exhibits higher stiffness when in the martensitic state. If the temperature is below martensite finish temperature (M_f), the NiTi alloy will be in the martensitic state which exhibits more softness and ductility and possesses the property of shape memory [6]. Consequently, heat treatment is known to modify the phase transition leading to more martensitic phase during treatment that would improve the mechanical performance of NiTi endodontic files with enhanced flexibility and fatigue resistance [7–9]. RaCe files (FKG Dentaire, La Chaux-de-Fonds, Switzerland) are conventional austenitic files that are electrochemically polished. They have triangular cross-sections with alternating cutting edges to reduce the screwing-in effect. RaCe EVO files were introduced as a newer version that have the same design and geometry, with the difference that they are heat treated. Both RaCe and RaCe EVO are used in continuous rotation.

Since a file's cyclic fatigue resistance is enhanced in a reduced ambient temperature, the current study aimed at evaluating the influence of short-term cooling by a refrigerant spray on the cyclic fatigue resistance of RaCe and RaCe EVO files within a dynamic cyclic fatigue model. Few previously published studies evaluated the effect of reducing the ambient temperature of the NiTi files on their cyclic fatigue resistance; however, none of them was done on NiTi files with the same design and geometry but with different metallurgy.

The null hypothesis tested in this study was that the cooling treatment would have no significant effect on cyclic fatigue resistance of the evaluated endodontic files under simulated body temperature.

3. Materials and methods

Forty-five NiTi files from RaCe and RaCe EVO (FKG Dentaire, La Chaux-de-Fonds, Switzerland) were used in this study with total of 90 files. All the files were of the same length (25 mm) and their specifications were Size 25 Taper 0.06. They were inspected under a dental operating microscope for deformities (Zumax OMS2350, Suzhou, China) at 30 \times magnification. Each group was subdivided into three subgroups ($n = 15$) based on the cooling application time. The first subgroup was tested as received. The second and third subgroups were tested after 5- and

15-seconds cooling application, respectively. Immediately after each cooling application, the dynamic cyclic fatigue testing was conducted.

The protocol followed for short-term cooling was that the file was mounted in the rotary handpiece (Dentsply Maillefer, Ballaigues, Switzerland), and while keeping the nozzle of the refrigerant spray (Endo Ice, Maquira Industria DA, Brazil) in contact with the file, it was sprayed back and forth along the functional part alone, for either 5 ± 1 or 15 ± 1 seconds, immediately preceding the commencement of its rotation.

The cyclic fatigue test was conducted by powering the rotary handpiece with a torque-controlled electric motor (X-smart plus, Dentsply Maillefer, Ballaigues, Switzerland) to rotate the files within an artificial curved canal with a curvature of 60° and 5 mm radius, with the entire apparatus fixed inside a water bath (DXY, Hong Kong, China) to maintain the temperature at 37 ± 1 °C. Each file was introduced into the artificial canal up to its end, and according to the manufacturer's instructions, they were powered at 1.5 N.cm torque and 800 rpm speed to rotate freely inside the canal with a controlled in-and-out motions. To obtain a reproducible simulation of dynamic axial motion, a dynamic test device consisting of a reciprocating cycle linear DC gear electric motor (Mainland, Guangzhou, China) fixed on a portable tripod (Promate, Shenzhen, China) was used. Silicone lubricant (CRC Inc., Warminster, VA, USA) was introduced into the canal to reduce friction and heat production. A video recording device was used to record the process for each file to be able to accurately record both the visual breakage of the instrument as well as its corresponding time. The instruments were rotated until a fracture occurred visually and/or audibly, and the number of cycles to failure (N_f) was calculated by multiplying the number of seconds in rotation by the speed of rotation of the motor (rpm)/60 seconds. Three fractured files from each subgroup were randomly selected, cleaned of debris in an ultrasonic cleaner for 6 min using 30% ethanol, vertically mounted using a custom holder and scanned using a Scanning Electron Microscope device (Jeol JSM-6610LV, Tokyo, Japan) at a 250 \times magnification using 10 kV, to examine the topography of the fracture patterns.

4. Statistical analysis

Sample Power Calculation was performed using the G-Power sample power calculator (Universtat-Kiel, Kiel, Germany). It was determined that for a one-way ANOVA distribution with an effect size of 0.05 and sample power of 0.95 with an alpha of 0.05 and 6 groups, we would need a total of 90 samples.

One-way ANOVA with Scheffe's post hoc test were used to analyze the N_f results. The level of significance was set at 5%. All statistical analyses were performed using the SPSS ver. 25 (IBM Corp., Armonk, NY, USA).

5. Results

Significant differences in the Nf between the tested subgroups were found ($p < 0.05$) (Table 1) at each testing condition, where RaCe EVO files showed improved cyclic fatigue resistance. There was a significantly higher Nf for the RaCe Evo files in all three subgroups (control, 5 sec cooling and 15 sec cooling) compared to the RaCe files. When the impact of cooling was compared for each type of file, it was observed that there were significant differences in Nf between the cooling times for the RaCe files but not for the RaCe Evo files (Table 2).

Table 1. Comparison of Nf among all groups.

	N	Mean of Nf	Std. deviation	F*	Sig
Race-No Cooling	15	633.7533	159.19311		
Race-5 sec Cooling	15	864.8800	119.14381		
Race-15 sec Cooling	15	753.7733	111.42105		
Race Evo-No Cooling	15	2213.3400	453.21581	95.546	<0.001**
Race Evo-5 sec Cooling	15	2317.1286	551.22224		
Race Evo-15 sec Cooling	15	2261.6688	324.33329		
Total	90	1506.8078	828.21558		

*Calculated using One-Way ANOVA.

**Differences significant at $p < 0.05$.

The data for the RaCe files were then subject to a Scheffe's post hoc test to examine the impact of cooling time. It was observed that cooling for 5 seconds had significantly greater Nf than the control group ($p < 0.001$). Files cooled for 15 seconds did not show a significant difference in Nf either with the control group ($p = 0.055$) or with the group cooled for 5 seconds ($p = 0.081$). The lengths of fractured segment were reported to be comparable in RaCe and RaCe EVO groups with 3.24 ± 0.80 and 3.09 ± 0.66 mm, respectively ($p > 0.05$).

SEM images revealed typical features of cyclic fatigue behavior in both systems (Fig. 1). The cyclic fatigue caused crack initiation at the cutting edges of the fracture cross sections, with an area of microscopic dimples and crack propagation characterized by striations.

6. Discussion

The current study confirmed that heat-treated files (RaCe EVO) were not affected by cooling treatment. RaCe files showed significant resistance to cyclic fatigue when cooled for 5-seconds compared to the other tested subgroups within the group of RaCe. According to Meling and Odegaard [10], short heating of an activated superelastic NiTi wire showed a small and temporary rise in bending stiffness, while when it subjected to short cooling, the bending stiffness decreased markedly for longer time, so the wire will be more flexible. The reasoning behind this,

is that the superelastic NiTi wires are primarily austenitic at body temperature when no force is applied, and when the wire is bent, the NiTi alloy is incompletely transformed from austenite to martensite phase. When exposed to cold, the alloy's temperature will be under its Ms temperature and is transformed into the martensitic form, and since martensite is more flexible than austenite, the stress for a given strain is reduced.

It has been reported that clinically the intracanal temperature at which the files are used is around 35°C [11]. The intracanal temperature has a drastic effect on the cyclic fatigue resistance of conventional nickel-titanium files as they are in the austenite phase at body temperature. On the other hand, the intracanal temperature does not influence the fatigue life of heat-treated files as their Af temperature is higher than that of the body temperature, and therefore they display a martensitic behavior at body temperature [12]. On the contrary, the cooling effect can allow for greater flexibility of NiTi [13], as the cooling of conventional NiTi files will reduce their temperature lower than the Af temperature, so the file will behave as a martensitic alloy. Interestingly, the results of the present study support those obtained by a previous investigation, where investigators evaluated the cyclic fatigue of different files at two different environmental temperatures: $20^\circ\text{C} (\pm 2^\circ\text{C})$ for room temperature group and $-20^\circ\text{C} (\pm 2^\circ\text{C})$ for the cooled environment group. Nf values were significantly higher for files from the cooled environment than for those from the room temperature group [13].

When assessing the cyclic fatigue resistance of the files, the use of standardized artificial stainless-steel canals has been recommended to minimize the variables arising from natural teeth [14]. In this study, the artificial stainless steel canal was produced according to Pruett *et al.* [15], where two parameters (angle & radius of curvature) were used to simulate the degree of canal curvature accurately.

Cyclic fatigue testing can be accomplished through static models, in which a file is flexed and then rotated until fracture occurs [16]. However, Dederich & Zakariassen [17], used a dynamic model and found that a cyclic axial motion significantly extended the life span of rotary files. In a dynamic model, the increase in forward-rearward motion in the axial direction provides the file with more time before passing high-stress zone [18]. This type of test more closely approximates a clinical brushing or pecking motion [19]. To simulate the clinical setting, the current study used a dynamic test model at a body temperature (37°C). Some authors have shown that body temperature drastically affects the flexural resistance of NiTi files because it is capable of modifying the transformation temperatures of the NiTi [20, 21].

The selection of 5- and 15-seconds cooling periods was decided upon after a pilot study was conducted to select the most appropriate duration for cooling treatment; wherein the authors found that applying a cold spray for 5 &

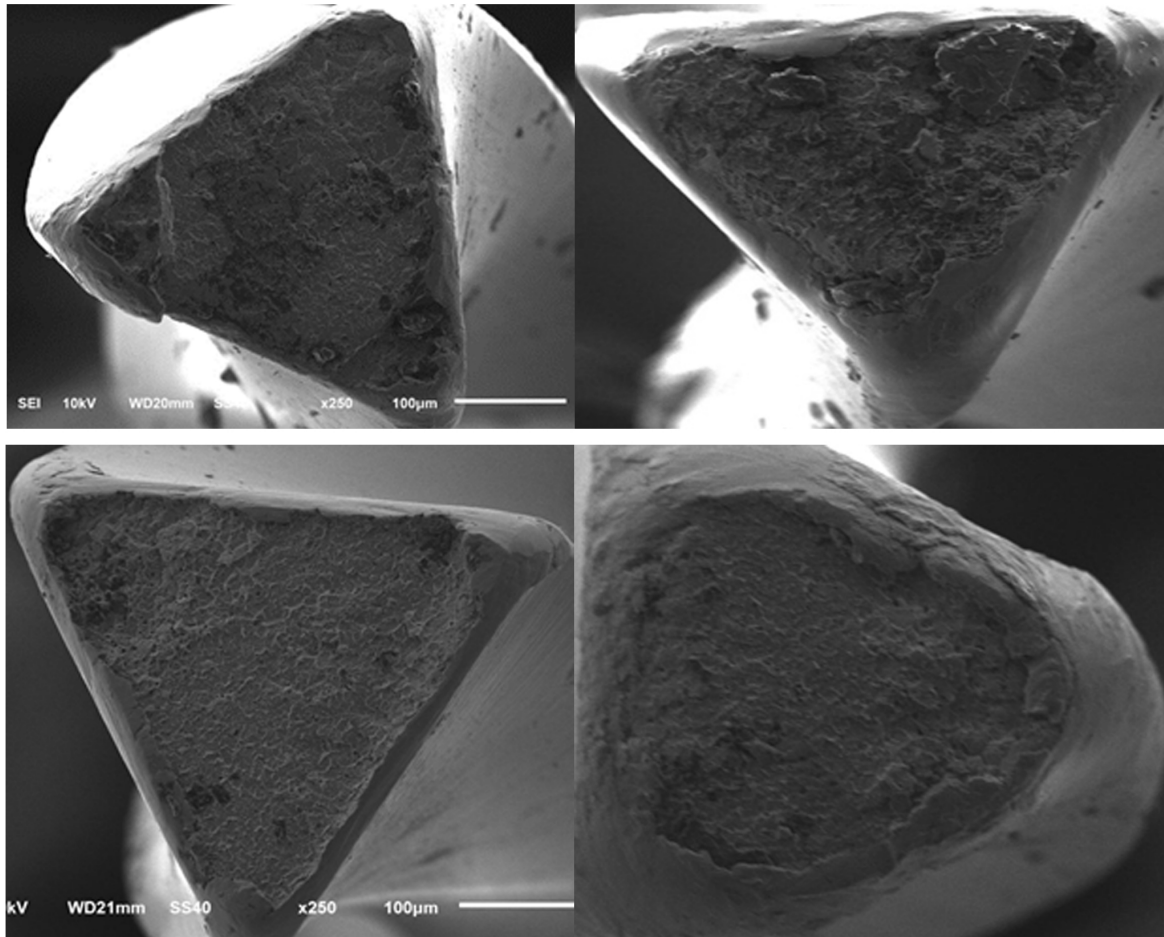


Fig. 1. Scanning electron microscopic images showing the fractured surfaces of RaCe and RaCe EVO after cyclic fatigue, confirming a predominantly ductile mode of fracture. Propagation points at the surface, striations and dimples are obvious on the surface.

Table 2. Impact of cooling time on Nf in each file type.

File		N	Mean of Nf	Std. deviation	F*	Sig
Race	Control (No Cooling)	15	633.7533	159.19311	11.573	<0.001**
	Cooling for 5 Seconds	15	864.8800	119.14381		
	Cooling For 15 seconds	15	753.7733	111.42105		
Race Evo	Control (No Cooling)	15	2213.3400	453.21581	0.153	0.861
	Cooling for 5 Seconds	15	2300.4333	535.09218		
	Cooling For 15 seconds	15	2274.6667	331.37530		

*Calculated using One-Way ANOVA.

**Differences significant at $p < 0.05$.

15 seconds led to a decrease of the file's temperature on average of -20.67 and -31.23 °C, respectively. Digital laser infrared thermometer (Laser Grip 1080, Etekcity, Anaheim, CA, USA) was used to measure the temperature of the files after cooling.

A major drawback of laboratory testing the fatigue behavior of NiTi rotary files is the inability to eliminate several confounding factors, such as material properties, design, and dimensions of the file, which are specific to the brand(s) being tested. This makes it difficult to quantify the effect of a single variable on fatigue behavior [22]. In

the present study, except for metallurgical differences, all other variables which could affect the results were eliminated. According to the manufacture, the RaCe EVO system is an updated version of the original system (RaCe), where no changes have been made in the cross-section, geometry, and size of the file. The difference between the two is that RaCe EVO has undergone a complex heating-cooling treatment that results in a visible blue titanium oxide layer on the surface of the file. The manufacturers claim that this thermal treatment controls the transition temperatures of alloys, and it increases the so-called austenite start (As) and

austenite finish (A_f) temperatures, which indicate the temperatures when the transformation of the metallic structure from martensite to austenite starts and is completed. When the temperature is between A_s and A_f , the alloy consists of austenite and martensite, which has higher fatigue resistance [23]. When the alloy cools down to a temperature close to A_s , the percentage of the martensitic phase could reach the maximum range possible for the alloy itself [24]. Nitinol file with more martensite phase has more flexibility, consequently, more resistant to fracture than austenite phase [6]. The results of the present study revealed that thermal processing during file manufacturing has a significant effect on cyclic fatigue resistance. RaCe EVO files have a significant increase in the resistance of cyclic fatigue than the non-heat-treated RaCe files regardless of cooling treatment time. The reason for this is that heat treatment of a NiTi wire increases the transformation temperature of the wire, so the austenitic temperature (A_f) of a heat-treated wire is higher than body temperature. Thus, the wire behaves as a martensitic wire at body temperature with enhanced mechanical properties than the non-heated wire [25]. This result was confirmed by Ugur *et al.* [26], who showed that N_f values of Reciproc (non-heat-treated) were significantly lower than Reciproc Blue (heat-treated) files. Another investigation [27] also showed a similar result; Reciproc Blue files were associated with higher cyclic fatigue resistance than the non-heat-treated files. Moreover, similar results have also been found in relation to torsional stress [28].

The artificial canals were made with dimensions similar to those of the tested files to eliminate the slight changes in file positioning which can affect the cyclic fatigue behavior. Consistent with previous studies, the fractured site was found to be close to the maximum curvature area; in the range of 3.09–3.24 mm from the file tip, showing that the point of maximum stress was similar in each condition. This confirmed the file positioning in a precise trajectory [29].

This study found that the cooling treatment of heat-treated files (RaCe EVO) has no significant effect on cyclic fatigue resistance regardless of cooling duration. This could be attributed to the lower percentage of a metal structure that may pass from austenite to martensite [13] as RaCe EVO alloy already contains a higher percentage of martensite phase. In addition, the A_f temperature of heat-treated files such as RaCe EVO is usually higher than the ambient temperature (37 °C) [22]; and therefore, there was no phase transformation. On the other hand, the group of RaCe files cooled for 5 seconds showed significantly ($p < 0.05$) higher resistance to cyclic fatigue compared to the control group. RaCe files are in the conventional (austenite) phase at body temperature [22]; therefore, they have more percentage of a metal structure that may pass from austenite to martensite when cooled. There was an insignificant increase in cyclic fatigue resistance of RaCe files when cooled

for 15 seconds compared to control and 5 seconds cooling. The potential reason behind this is that prolonged cooling may lead to a decrease of the file's temperature beyond a plateau state, where the metal structure completely transformed to martensite; thus, the metal lost its original properties of super elasticity and shape memory, to become a stiff metal. Consequently, the cooling treatment did not enhance the flexibility of the metal beyond this plateau point [30]. Based on the results of the present study, the null hypothesis of the study was rejected as there was a significant difference between cyclic fatigue resistance of RaCe files after cooling.

The results of studies that use artificial canals must be extrapolated to clinical conditions with caution because of the differences between a stainless-steel block and dentin [31] and is considered as a limitation in this study. Moreover, the use of Micro-X-ray diffraction and temperature-modulated differential scanning calorimetry (DSC) to investigate microstructural phases, phase transformations, and effects of heat treatment for rotary nickel-titanium instruments are recommended for future studies.

7. Conclusions

Within the limitation of the present study, it can be reconfirmed that thermal processing during the manufacturing of NiTi file has the most influential effect on the mechanical wire's properties. Short-term cooling application for 5 seconds using a refrigerant spray on RaCe NiTi files (superelastic) enhances its cyclic fatigue resistance significantly. On the other hand, it had no effect on the cyclic fatigue resistance of RaCe EVO NiTi files (heat-treated). Thus, cooling superelastic files for 5 seconds using a refrigerant spray ahead of use might be recommended for cases at high risk of file separation due to flexural failure such as curved canals.

8. Author contributions

AA and MA equally participated in the design, experiment doing, analysis, and writing the manuscript.

9. Ethics approval and consent to participate

Authority to conduct the research was sought from the Riyadh Elm University Research Center Ethics and Standard Committee, registration No. FPGRP/2020/503IRB and approval No. FPGRP/2020/503/283.

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12. Conflict of interest

The authors declare no conflict of interest.

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