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Cyclic fatigue testing of Three NiTi files at different temperatures and their corresponding phase transformations (In vitro study)

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Abstract

Objective: To compare between Revo-S, ProTaper Next and M3 ProGold files regarding Cyclic fatigue resistance at Room temperature and at Intracanal temperature with the corresponding phase transformations for each instrument at these temperatures.

Materials and methods: 1) Instrument cyclic fatigue: Twenty files 25/0.06 of each system were used. Ten of them were tested in room temperature and ten in Intracanal temperature. Cyclic fatigue testing of instruments at room and intracanal temperature was performed with dynamic cyclic fatigue testing device. The number of cycles to failure (NCF) was calculated. 2) Differential Scanning Calorimetry (DSC): Three to five mm of Revo-s, PTN and MPG files was used. The DSC analyses was conducted over a temperature ranging from 0 to 50 ^{o}C .

Results: The MPG system showed significantly the highest cyclic fatigue resistance values followed by PTN then Revo-S in both room and intracanal temperature. It was found also that there is reduction in cyclic fatigue resistance of all file systems when testing in intracanal temperature rather than room temperature. The DSC confirmed the presence of martensitic phase in MPG system at both room and intracanal temperature with more martensitic percentage at room temperature

Conclusion: files made from CM wire (MPG) would provide better cyclic fatigue resistance values rather than files from M wire (PTN) or Austenitic wire (Revo-S) at

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both room and intracanal temperatures.

1.Introduction

NiTi wires were introduced to use for root canal preparation which was considered a breakthrough in root canal preparation. The exceptional properties of NiTi files including shape memory, super elasticity and flexibility made their use very promising. The main problem with NiTi files was the increased liability to separation with no previous alert. The modes of failure that affected NiTi files were mainly cyclic fatigue failure, torsional failure or combination of both. Cyclic fatigue failure is caused by successive compressive and tensile stresses on file during rotating inside root canal. It was found that failure that occurred was due to many factors including the operator, the tooth anatomy, NiTi files' design and metallurgy. NiTi files underwent various manufacturing improvements in both design and metallurgy to overcome or reduce these modes of failure. The three files chosen for this study were files with great popularity among clinicians either due to their innovative design or metallurgical improvements .

Materials and methods

1-cyclic fatigue resistance:

Sample selection: A total of 60 NiTi rotary files of the three brands were used; Revo-S"SU", Protaper Next "X2"& M3ProGold"MPG3", (twenty file of each brand). All with ISO tip size #25 (0.25mm) and taper 6%. All instruments were inspected under digital microscope with 8X magnification for any signs of visible deformation and the defective instruments were discarded**. Sample size calculation:** Number of samples was chosen to be 10 for each subgroup. This was in accordance with the recommendations of ASTM International [1] **.Sample classification:** Files used in this study were classified into three main groups (each n=20) according to the file's brand:

- Group I (Revo-S group): Revo-S files size #25 (0.25mm) and 6% constant taper.
- Group II (ProtaperNext "PTN" group): ProtaperNext files size #25 (0.25mm) and 6% variable taper.
- Group III (M3proGold"MPG" group): M3proGold files size #25 (0.25mm) and 6% constant taper.
- Each group was subdivided into 2 subgroups according to temperature under which test was performed (n=10):
- Subgroup a (in Room temperature) 22 ± 0.5 °C
- Subgroup b (in Intracanal "IC" temperature) 35 ± 0.5 °C

Cyclic fatigue resistance

The custom-made dynamic cyclic fatigue-testing device designed and assembled by M.El-Wakeel was used. The artificial canal was designed according to the technique proposed first by Grande et al., [2] in 2005. The canal was designed to have the same size and taper of the files to be used (25/0.06) plus 0.1 mm relief circumferentially for the whole file. The canal has 60° curve with 3 mm radius of curvature, Maximum convexity is 5 mm from the tip of the canal. The canal is not a perfect canal; it is rather a groove in a metal block. There is a missing side of the canal which is important to allow monitoring of files during testing and easy replacement of files between experiments. This missing side is reestablished by a piece of glass fixed in place by two hand screws .this groove will be referred to as the artificial canal throughout the paper. A cylindrical space with 5 mm radius is added to the canal tip to allow an easy escape to broken fragments. This would ease observation of the tests and replacement of broken files. The testing device is adjusted so that the file enters the canal in its center and the file tip coincides with the canal tip. Each file is attached to the endodontic motor E-cube (Saeshin precision, Korea) with 16:1 reduction hand piece. The motor settings were adjusted having the recommended rpm for each file brand (with auto-reverse function disabled).The artificial canal is flooded with lubricating oil .The file is introduced into the canal and the glass cover is screwed. The endodontic motor, the dynamic movement regulator and the stopwatch are activated simultaneously. The dynamic movement regulator provided axial movement (up-down) that had an amplitude of 3 mm [4,5] .While the file is rotating in the canal, it is observed visually carefully by the operator until the file is broken. Time until fracture was recorded with digital stop watch accurate to 0.01 seconds. For testing in intracanal temperature, cyclic fatigue testing was done under water bath of distilled water at 35 ± 0.5° C simulating intracanal temperature [6,7] , by using water bath glass container. The temperature of the water was controlled via aquatic thermostat connected to heat control and measured via digital thermometer. The number of cycles to failure (NCF) was calculated by the following equation [8,9] : NCF = Time to fracture (seconds) × Revolutions per minute (rpm)/60.

2-Differential Scanning Calorimetry (DSC):

Five specimens from new rotary NiTi endodontic instruments, taper 0.06, size 25 and 25-mm long were examined: Revo-S"SU", PTN"X2", M3progold "MPG3" were evaluated.

Each test specimen was placed in an open aluminum pan. An empty aluminum pan served as the inert control specimen for the DSC measurements .Segments were sealed using a standard Sample Pan Crimper Press. The DSC analyses were conducted over a temperature ranging from 50ºC to 0ºC to obtain the cooling DSC curve, and subsequently heated from 0ºC back to 50ºC to obtain the heating DSC curve.The liner heating or cooling rate was a standard 10ºC/min and during each analysis the DSC cell was purged with dry nitrogen at a rate of 50mL/min. The Plots were analyzed by Universal V4.5A TA instruments computer software to obtain the onset temperatures for the phase transformations, along with the enthalpy changes (ΔH) associated with these processes. The transformation temperatures were obtained from the intersection between extrapolation of the baseline and maximum gradient line of the Lambda-type DSC curve. The martensitic transformation-starting and transformation-finishing points (M_s, M_f) and reverse transformation-starting and transformation-finishing points (A_s, A_f) were determined when found [10].

3.Statistical analysis:

After obtaining NCF for all files, data was statistically analyzed using SPSS software. Two way analysis of variance was performed. One way analysis of variance was performed to find the independent effect of each of the variables on cyclic fatigue resistance followed by Post Hoc test which was performed when P value ≤ 0.05 for pair wise comparison. All tests were run at 95% confidence interval and statistical significance was set at P value less than 5% (α =.05).

4.Results:

1-Cyclic fatigue resistance:

After the data were collected, tabulated and statistically analyzed, the results of the different tests showed the following: **Two way ANOVA** test showed that both type of file and temperature had statistically significant effect on cyclic fatigue resistance (NCF). However the interaction between the file type and temperature had no statistically significant effect on cyclic fatigue resistance (NCF).

One-way ANOVA followed by Tukey HSD post hoc test showed the following results.

At room temperature, It was found that the highest cyclic fatigue resistance was for MPG (414.62 ±40.7), followed by PTN (255.88 ±24.32), and the lowest was for Revo-S (131.06 ±11.22) with statistically significant difference between all results .While at **Intracanal (IC) Temperature ,** It was found that the highest cyclic fatigue resistance was for MPG (379.53 ±17.88), followed by PTN (248.28 ±23.95), and the lowest was for Revo-S (105.67 ±11.02) with statistically significant difference between all results. *Results showing the effect of Temperature on cyclic fatigue resistance (NCF)* of different file types showed that for **Revo-S,** results showed higher cyclic fatigue resistance (NCF) of Revo-S at room temperature

(131.06 ±11.22) than at IC temperature (105.67 ±11.02) with a statistically significant difference between them. **For PTN,_**results showed higher cyclic fatigue resistance (NCF) of PTN at room temperature (255.88 ±24.32) than at IC temperature (248.28 ±23.95) with no statistically significant difference between them.For MPG,results showed higher cyclic fatigue resistance (NCF) of MPG at room temperature (414.62 ±40.7) than at IC temperature (379.53 ±17.88).

with a statistically significant difference between them.

Differential scanning calorimetry

The DSC plots for both the heating and cooling cycles of different types of NiTi rotary instruments (Revo-S "SU" ,PTN "X2" , M3ProGold "MPG3") used in this study showed that the DSC curve for conventional superelastic rotary instrument Revo-S (SU) and for M-wire rotary instrument ProTaper Next (X2) exhibited no transformation neither on heating nor on cooling.

The DSC curve for CM-wire rotary instrument M3 Pro Gold (MPG3) exhibited a single endothermic peak on heating curve which represents the transformation from martensite to to austenite phase .The cooling curve showed a single peak which represents the phase transformation from austenite phase to martensite .

The Austenite-start temperature is 39.56 °C while the Austenite-finish temperature is (43.66°c) with total enthalpy "ΔH" 1.885 j/g for CM-wire rotary instrument (M3 Pro Gold) . The martensetic-start temperature is (37.16°C) while the martensetic –finish temperature is (34.49°C) with total enthalpy "ΔH" 1.945 j/g for CM-wire rotary instrument (M3 Pro Gold).

5.Discussion

Rotary NiTi files' separation remains a major concern during endodontic instrumentation as it is an unavoidable incident during clinical use [3,11]. Although multiple factors contribute to file separation, cyclic fatigue has been shown as one of the leading causes [12]. Fracture caused by fatigue through flexure occurs because of metal fatigue. The instrument does not bind in the canal, but it rotates freely in a curvature, generating tension/compression cycles at the point of maximum flexure until the fracture occurs [13,14] .

Several non-tooth devices [15] used to investigate in vitro cyclic fatigue resistance in both static or dynamic modes were reviewed. Dynamic cyclic fatigue testing was used in this study as dynamic movement of the file better simulates the stress that is generated on a file in a clinical situation as the maximum point of flexure varies along the file throughout the testing procedure [16].

Dederich & Zakariasen [17] demonstrated that an axial movement can significantly increase the resistance to cyclic fatigue of endodontic instruments. Ray et al. [18] also used dynamic study designs with a standardized axial movement. The axial movement in dynamic test designs prolonged the stress interval until cyclic fatigue occurred [17] . In the present study dynamic model was used while trying to maintain a precise trajectory during testing by using stainless steel block with artificial canal having size and taper closely resemble files tested with 0.1 mm relief circumferentially for the whole file to keep instrument running freely without torsional load and also by stabilizing the endodontic handpiece at the correct angulation allowing straight entry to the simulated canal using an endodontic motor mold and self-locking nylon cable zip ties.

Temperature has been reported as another important confounding factor in cyclic fatigue studies. It has been shown in several studies [19, 20, 21, 22, 23] that environmental temperature significantly influences the cyclic fatigue behavior of NiTi instruments. The metallurgy of NiTi alloys exhibits an eminently different behavior at room or at body temperature [24]. and in the present study the cyclic fatigue resistance was measured at both room temperature and intracanal temperature like in previous studies [6,7] which simulate the clinical conditions inside root canal even more than testing in body temperature. The phase transformations of the different NiTi alloys can be assessed using either differential scanning calorimetry (DSC) analysis or x-ray diffraction. In this study , DSC was used rather than other methods as it was found to be superior to x-ray diffraction which only determines phases in the superficial first 50 μm of the surface [(25)].These phase transformations would give an indication about metallurgy found in each file.

Results in this study showed that Revo-S (SU) had the lowest cyclic fatigue resistance (NCF) in both room and intracanal temperature followed by PTN (X2) then MPG (MPG3) which had the highest cyclic fatigue resistance. It was suggested that the results concerning cyclic fatigue resistance of NiTi instruments is influenced by various factors, including design, manufacturing method and the properties of raw materials.

Firstly, to emphasize the effect of design and manufacturing method on cyclic fatigue resistance from previous studies .Design of sharp and fine points of Revo-S have poorer stress distribution than those with a convex or triple-helix configuration even with the same metallurgy like one shape [(8,26)]. The lower NCF of PTN in comparison with MPG can be explained by some studies [(27,28)] which showed that NiTi instruments having a triangular cross-sectional geometry like MPG (convex triangle) provided better fatigue resistance than a square or rectangular cross-section like PTN.

Secondly ,the effect of change in material alloy or metallurgy was studied .Miyai et al. [(29)] indicated that the mechanical performance of NiTi endodontic instruments may be closely correlated with the transformation behavior of the alloy.

In the present study, it was found that Revo-S (SU) ,made from austenitic alloy, showed no phase transformation neither on heating or cooling which indicates that at temperature range of DSC in this study ranging from 0-50°C the Revo-S file would be completely in Austenitic form in both room or intracanal temperature . The M3 Pro Gold files, as CM wire, have a mixture of martensite and austenite at room temperature. In particular, the A_f of the MPG was measured to be 43.66 °C from the heating curve. This means that at intracanal temperature (around 35°C) [(30)] , the M3ProGold files are in a mixed austenite and martensite phase.. The DSC curve also can explain the significant difference in cyclic fatigue resistance of MPG between room and intracanal temperature as it is suggested to have less martensitic grains at intracanal temperature than the more martensitic grains found in room temperature.

6.Conclusion:

Under the circumstances of this study, it can be concluded that:

Different microstructure of NiTi files had a noticeable effect on cyclic fatigue resistance of NiTi files.

The effect of the rotary NiTi instruments' design cannot be neglected or under estimated.

The effect of temperature on cyclic fatigue resistance should be considered in the upcoming researches as it has a direct effect on cyclic fatigue resistance results.

7.References

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