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Effect of thermal treatment on the flexural resistance of mechanically pre-fatigued NiTi rotary endodontic files

Key words  flexural resistance, nickel titanium, thermal treatment

Introduction: The aim of this study was to determine the effect of thermal treatment on the flexural resistance of mechanically pre-fatigued rotary nickel-titanium (NiTi) endodontic instruments.

Methods: New and mechanically pre-fatigued rotary NiTi instruments were subjected to thermal treatments at 350°C, 400°C and 450°C for 30 minutes, and then were rotated in a fatigue-testing device until fracture. The time to fracture was recorded and multiplied by the number of rotations per minute to obtain the number of cycles to failure (NCF) for each group. One-way analysis of variance (ANOVA) followed by Tukey–Kramer multiple-comparison post hoc test was used for the comparison between the different tested groups. Fractographic examination by scanning electron microscopy was also performed to study the morphological characteristics of the fractured instruments.

Results: Statistical analysis showed that the thermal treatment at or below 400°C of the new files did not improve the resistance to the flexural fatigue. Also, the flexural fatigue resistance of mechanically pre-fatigued files was significantly lower than that of new files. However, with thermal treatment significant improvement in the resistance to flexural fatigue was achieved, especially at 450°C (P < 0.05).

Conclusion: Thermal treatment seems to be of significance in improving the cyclic fatigue resistance or life span of fatigued or used NiTi files.

Introduction

Root canal preparation using rotary nickel-titanium (NiTi) instruments with super-elastic properties often results in faster, more consistent preparations with fewer procedural errors. Despite these advantages, unexpected instrument fracture is not uncommon. Sattapan et al identified two modes of fracture for rotary NiTi instruments; torsional failure and flexural or cyclic fatigue. The former occurs when the tip or any part of the instrument locks into the canal while rotary motion continues, while the latter is attributed to work hardening and metal fatigue. It occurs at the point of maximum flexure when the instrument is freely rotating in a curved canal, and may originate from defects in the metal surface.

Of clinical concern is the fact that flexural fatigue occurs with no visible signs of plastic deformation, thus the clinician should be aware of the factors that contribute to this type of failure in order to prevent...
its occurrence. These factors\(^5\) include the anatomy of the root canal system both in terms of radius and degree of canal curvature, operating speed and torque, sterilisation procedures, previous use as well as the size, taper and characteristic design features of the instrument itself.

Another important factor is the thermomechanical processing history of the NiTi alloy throughout the manufacturing process. Manufacturing NiTi rotary instruments involves the machining of a wire blank, leaving a surface that is irregular, stressed, plastically deformed or contaminated\(^6\). Thus heat treatment during manufacturing is performed to release the internal strain caused by work hardening of the alloy\(^7\)-\(^9\). Also, electropolishing is sometimes done to reduce the machining damage on the file surface\(^10,11\). Previous studies\(^7,8,12-15\) have shown that additional thermal treatments significantly modify the mechanical and super-elastic properties of NiTi files. However, the effect of thermal treatment on rotary files that are mechanically fatigued inside root canals is still unknown. Therefore, the purpose of this study was to determine the effect of thermal treatment on the flexural resistance of mechanically pre-fatigued rotary NiTi endodontic instruments.

\section*{Materials and methods}

Eighty NiTi endodontic files (RaCe, FKG, La-Chaux De Fonds, Switzerland) of size 30, 0.04 taper and 2 mm length were used in this study. Electropolished files were obtained from the manufacturer from the same batch. Eighty instruments were used to assess the effect of thermal treatment on the flexural resistance of the files after mechanical fatigue as well as for fractographic analysis by scanning electron microscopy (SEM).

\section*{Classification of the samples}

The 80 samples were classified into eight equal groups (n = 10) in the following manner:

- Group 1 (NFC): As-received new files that were rotated in a fatigue-testing device until fracture in order to determine the mean time required for fracture (Tf).
- Group 2 (FC): Mechanically fatigued files that were rotated half the mean time required for fracture (1/2 Tf), bench rested for 1 hour and then rotated until fracture.
- Group 3 (NF350): New files that were heat treated at 350°C for 30 minutes in an electric furnace (Midetherm, BEGO, Germany) and then rotated until fracture.
- Group 4 (NF400): New files that were heat treated at 400°C for 30 minutes and then rotated until fracture.
- Group 5 (NF450): New files that were heat treated at 450°C for 30 minutes and then rotated until fracture.
- Group 6 (F350): Files that were mechanically fatigued (1/2 Tf), then heat treated at 350°C for 30 minutes and then rotated until fracture.
- Group 7 (F400): Files were mechanically fatigued (1/2 Tf), then heat treated at 400°C for 30 minutes and then rotated until fracture.
- Group 8 (F450): Files were mechanically fatigued (1/2 Tf), then heat treated at 450°C for 30 minutes and then rotated until fracture.

\section*{Fatigue testing}

The fatigue-testing device used for this study consisted of a stainless steel block (25 mm x 25 mm x 40 mm) with a simulated canal that was milled with a precision milling machine. The artificial canal had an angle of curvature of 60 degrees and radius of curvature of 5 mm. The centre of the curvature was approximately 5 mm from the tip of the instrument, the curved segment of the canal was approximately 5 mm in length and the linear segment between the tip of the instrument and the end-point of the curvature was approximately 2.5 m. The artificial canal was constructed with a tapered shape corresponding to the dimensions of the instruments tested according to the method proposed by Plotino et al\(^16\).

Eight blocks were constructed; one for each group. The block was fixed to the lower compartment of the Universal Testing Machine (Lloyd Instruments LR5 series, Bognor Regis, UK) while the endodontic handpiece with the rotating NiTi file was mounted on the upper compartment. The instru-
ments were rotated at a constant speed of 300 rpm using a 16:1 reduction handpiece (Anthogyr, Sal-\textlanches, France) powered by a torque-controlled electric motor (E-Go, Sweden & Martina, Padova, Italy). To reduce the friction of the file as it contacted the artificial canal walls, special high-flow synthetic oil designed for lubrication of mechanical parts (Super Oil, Singer Co. Ltd, Elizabethport, NJ, USA) was applied. After accurate file positioning to its full length, it was set to rotate freely synchronised with timing by a digital stopwatch (Timex, Middlebury, CT, USA) to the thousandth of a second. Timing was stopped as fracture was detected visually and audibly. The time to fracture was recorded and the number of rotations until fracture was calculated to the nearest full number for each group. The time to fracture was multiplied by the number of rotations per minute to obtain the number of cycles to failure (NCF) for each instrument. The NCF data was expressed as mean ± standard deviation (SD) and was analysed by WinSTAT v. 2007.1. One-way analysis of variance (ANOVA) followed by Tukey–Kramer multiple-comparison post hoc test was used for the comparison between the different tested groups. \( P \leq 0.05 \) was considered significant.

### SEM analysis

Three fractured instruments were randomly selected from each group. They were ultrasonically cleaned in absolute alcohol for approximately 120 seconds and then mounted with the fracture end facing upward for fractographic examination by SEM (Philips SEM 515, Eindhoven, the Netherlands) to determine the morphological characteristics of the fractured instruments.

### Results

### Flexural resistance

The results of the statistical analysis of the NCF data are presented in Table 1. The statistical analysis showed that the thermal treatment at or below 400°C of the new non-mechanically pre-fatigued files did not improve the resistance to the flexural fatigue. In addition, the resistance to cyclic fatigue of mechanically pre-fatigued files is significantly lower than of new as-received file. However, with thermal treatment significant improvement in the resistance to flexural fatigue was achieved, especially at 450°C.

### Fractographic analysis

The appearance of the fractured surfaces, assessed by SEM, showed similar fracture characteristics for all groups and indicated that breakage of instruments was the result of fatigue. At low magnification (Fig 1), the fracture surface revealed a relatively flat and smooth fatigue zone at the periphery where fatigue crack propagation occurred in response to cyclic stresses, and a central rougher overload zone representing the site of the final catastrophic failure. At high magnification (Fig 2), the overload zone revealed evidence of dimpled rupture that is characteristic of the ductile nature of the ultimate catastrophic failure.

### Discussion

NiTi instruments with pseudoelastic properties (shape memory effect and super-elasticity) have been used to avoid or to limit the failure risk during endodontic treatment. However, cyclic deformation during use changes their mechanical behaviour and may lead to fatigue failure\(^8\). The super-elastic behaviour of NiTi instruments is associated with a reversible stress-induced transformation of the parent b-phase, austenite (B2 cubic crystal structure) from...
which the NiTi wire blanks are produced, to martensite (monoclinic B19¢ structure), which enables a large recoverable elastic strain. This behaviour is affected by three major factors: the first is the chemical composition of the alloy, the second is the method of fabrication and the third is the thermal history of the alloy.

Thermomechanical processing of NiTi shape memory alloys is known to strongly influence their mechanical properties as well as the various phase transformation temperatures. During cold working, the material is subjected to deformation or stress by machining a high density of lattice defects or dislocations. Although the presence of dislocations in a crystalline material such as an alloy is essential for plastic deformation, the overgrowth of dislocation density induced by cold working has the inverse effect, decreasing the ductility of the alloy. This is in addition to the fact that each dislocation produces a strain field, hindering the sliding of adjacent dislocations.

Annealing through thermal treatment gives the atoms enough thermal energy to rearrange themselves in the lattice. This is done during manufacturing to release internal strains from processed files and upgrade their mechanical properties. Cold worked NiTi alloys designed to be superelastic must be heat-treated at relatively low temperatures, in order to promote partial recovery of the deformed microstructure and the precipitation of the intermediate phase Ti3Ni4, which favours the formation of the R-phase, another martensitic phase whose presence in the alloy improves super-elasticity. A question then arises, would additional thermal treatment affect the mechanical properties of used NiTi rotary files? Thus the aim of the present work was to study the effect of thermal treatment on the flexural resistance of mechanically pre-fatigued NiTi rotary files.

RaCe instruments were selected for use in the present study as they exhibit adequate flexural fatigue resistance, probably because of a lower flexural rigidity for its cross section and the near absence of machining marks after electropolishing. Modified cross sections that offer increased cutting efficiency in addition to reduced contact areas (no or relieved radial lands) with the canal walls, were shown to reduce the magnitude of internal stresses generated inside the instrument. A relatively large sized instrument with an increased taper (#30/0.04) was selected. It is well known that the larger the metal volume of the instrument, the lower its flexural resistance.

The dimensions of the simulated canal in depth and width were based on measurements of the tested file to ensure an accurate trajectory during fatigue testing. Plotino et al. found that if the simulated canal does not simulate the size and shape of the instrument, the curvature placed on the instrument will be reduced, thus influencing the results of the test. Although the simulated canal does not duplicate the in vivo situation, it allows for the comparative testing of different instruments in a standardised environment.

The simulated canal used in this study standardised...
the working environment of each instrument. This was validated by the fact that all of the instruments tested almost fractured at the same point.

The fatigue testing device used in this study was mounted in a similar way as in Larsen et al25. Engraved marks on the Instron machine were used for ensuring reproducible precise three-dimensional alignments and positioning of the instruments. The simulated canal was initially covered with a tempered glass cover to prevent the instruments from slipping out. However the trajectory was so precise, it was not used.

During selection of the thermal treatment range, the range 350°C to 450°C was selected because previous studies reported that below 300°C the influence of heat treatment temperature is not sufficient to release crystal lattice defects26, while above 450°C there is a tendency for a reduction in the ductility of the heated alloy due to the precipitation of Ti3Ni4 which hinders dislocation motion13. With respect to the heat treatment period, a 30 minute period was selected. This is in agreement with Zinelis et al14 and Yahata et al27.

Results of the study showed that the thermal treatment at or below 400°C of the new non-mechanically pre-fatigued files did not improve the flexural resistance. This is probably because these files underwent the appropriate thermal treatment during manufacturing. However, thermal treatment at 450°C resulted in a significant increase in flexural resistance. This might be due to the larger thermal energy imparted to the alloy with resultant increased ductility. The resistance to cyclic fatigue of mechanically pre-fatigued files was found to be significantly lower than that of new as-received files. This was expected, as used files build up cumulative stresses that hinder dislocation motion and make them susceptible to fatigue failure.

Also, an important finding was that the thermal treatment significantly improved flexural resistance of mechanically pre-fatigued files, especially at 450°C. The explanation of this behaviour is due to the annihilation of lattice dislocations. This is in agreement with Kuhn and Jordan8 who suggested thermal treatment (recovery) at 400°C before machining of files to decrease the work hardening of the alloy, and Zinelis et al14 who found that the fatigue resistance of files steadily increased from the as-received state to 440°C annealing temperature. Another possible explanation is the presence of a hybrid microstructure that has an improved resistance against fatigue crack initiation as well as a favourable crack propagation resistance28.

It is worth mentioning that thermal treatment has an effect on the characteristic transformation temperatures of the heated alloy, and also on its cutting ability and/or hardness, however the evaluation of these changes is beyond the aim of this study. Also, the results cannot be extrapolated to other commercially available rotary NiTi endodontic instruments due to differences in geometric features, as well as in the thermomechanical history of the NiTi alloy.

Regarding the fractographic analysis, the relatively small fatigue zones that accompanied the large overload zones suggest that most of the fatigue lives of the used instruments were spent in the crack initiation phase. However, once cracks were initiated, they propagated rapidly, creating heavy stress concentrations that rapidly spread inward and quickly led to failure along the centre of the instrument shafts29. On the other hand, the ultimate failure of the instruments occurred due to the coalescence of the microvoids in the overload zone30.

Within the limitations of this study, it can be concluded that thermal treatment seems to be of significance in improving the cyclic fatigue resistance or life span of fatigued or used NiTi files.

References