

Original Article

Effect of deep cryogenic treatment on cyclic fatigue resistance of controlled memory wire nickel-titanium rotary instruments

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ABSTRACT

Background: Cryogenic methods have been used to increase the wear, abrasion, corrosion resistance and to improve the strength of metals. The aim of this study was to evaluate the effect of cryogenic treatment on the cyclic fatigue resistance of rotary nickel-titanium (NiTi) instruments.

Materials and Methods: In this *in vitro* study, 20 Hyflex (Coltene, Altstätten, Switzerland) nickel-titanium instruments, size 25, 0.06 taper, were randomly divided into two groups as follows: untreated NiTi rotary files (Group A) and cryotreated NiTi rotary files (Group B). The instruments of Group A were completely immersed in a cryocan containing liquid nitrogen (-196°C) for 24 h. After 24 h, the instruments were removed from the bath and were allowed to return to room temperature gradually. All files were used (at a speed of 500 rpm and a torque of 2.5 Ncm) in an artificial canal with a 60° curvature until fracture. Time to failure was recorded with a stopwatch in seconds and subsequently converted to number of cycles to fracture (NCF). Groups were compared using the independent-samples *t*-test. The level of statistical significance was set at 0.05.

Results: Mean NCFs in Group A and B were 1576 and 1395, respectively. However, statistical analysis showed that there was no significant difference between the groups ($P = 0.2$).

Conclusion: Deep cryotherapy of NiTi endodontic files cannot improve the cyclic fatigue resistance of HyFlex files, but further studies are required to evaluate these cryogenically treated NiTi files clinically.

Key Words: Cryotherapy, dental instruments, endodontics

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INTRODUCTION

Biomechanical preparation has been suggested to play the most critical role in root canal therapy for which nickel-titanium (NiTi) rotary instruments are among the most important instruments ever.^[1] Ideally, a NiTi rotary instruments should demonstrate sufficient flexibility and cutting efficiency. Such qualities prevent the file fracture within the canal and preserve the initial form of the canal.^[2]

In 1988, Nitinol (NiTi), an orthodontic wire alloy appeared in the endodontic literature.^[3] The alloy possesses matchless properties in terms of shape memory and pseudoelasticity that allow the development of rotary instruments. In comparison with stainless steel instruments, those of NiTi enjoy significantly higher flexibility and resistance to torsional fractures.^[3]

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Although beneficial, NiTi instruments have been frequently reported to fracture within the root canal system during endodontic treatment without any indication. When applied in curved canals, endodontic instruments undergo both tensile and compressive stress during rotation. Flexural fatigue, regarded as the most destructive cyclic fatigue, is owing to the frequent bending and unbending, encouraged during the use of endodontic instruments in curved canals. This is localized at the point of curvature. Research suggest that constant practice of tensile and compressive forces could be a source of a potentially destructive load in the area of curvature of the canal where NiTi rotary instruments are in use.^[4]

Cryogenic methods have been in practice to both enhance the level of wear, abrasion, corrosion resistance, and reinforce the metals. When treated cryogenically, NiTi instruments have boosted the degree of microhardness.^[4]

Cryogenic treatment (CT) is a two-stage process: (1) submersing the metal in a super-cooled bath that contains liquid nitrogen ($-196^{\circ}\text{C}/-320^{\circ}\text{F}$) and (2) allowing the metal to slowly let the cold out to reach room temperature.^[5]

Based on the treatment temperature, CT falls into shallow and deep categories.^[6] Conventional subzero treatments have been in use at approximately -80°C (shallow).^[7] However, the tool life grows at lower temperatures (deep) induced by liquid nitrogen at -185°C and -196°C .^[8,9] The process is labeled as wet as the material is immersed in liquid nitrogen, whereas in a dry process the material is just held at a position above the level of liquid nitrogen.^[2]

Cryogenic is a cost-effective treatment. Contrary to surface treatment techniques, CT could entirely affect the cross-section of the metal rather than a limited impact on the surface.^[5]

The effect of CT on the mechanical behavior of Ni-Ti instruments made of a recently established Ni-Ti alloy called control memory (CM) wire (CM) has not been evaluated so far in the literature.

CM wire is subjected to sophisticated thermomechanical processes (cooling and heating treatment), making the files extremely flexible, yet it lacks the property of preserving the shape memory in contrast with other conventional NiTi instruments.^[10]

As a result, the present study investigated the impact of deep wet CT on the cyclic fatigue resistance of a rotary file with a CM wire alloy.

Its null hypotheses considered that, there are no differences between cyclic fatigue of files treated or not treated with deep wet CT.

MATERIALS AND METHODS

Twenty Hyflex CM files (Coltore Hyflex, Altstätten, Switzerland) size 25, 0.06 taper were used in this *in vitro* study. These files were randomly assigned into two groups to examine the cyclic fatigue:

Group A (control group): Ni-Ti files which were not subjected to CT and Group B (study group): Ni-Ti files which were subjected to CT.

Cryogenic treatment protocol

The study group instruments were cryogenically treated with liquid nitrogen. They were completely immersed in a cryocan containing liquid nitrogen (at -185°C – -196°C) for 24 h [Figure 1]. Then, being removed from the bath, they were stored in 70% of absolute alcohol (Medicare, Montreal, Canada) to gradually return to room temperature. The protocol was similar to the one Singh *et al.* used in their study.^[11]

Protocol of cyclic fatigue examination

The device, developed in the present study to examine the strength of cyclic fatigue, include a main frame with fixed plastic support to hold the handpiece and a block made of stainless steel with a curved canal (with a radius of 5 mm and an angle of 60°), both installed on the frame. The canal was designed in accordance with the Hyflex file characteristics (size 25, 0.06 taper). Thus, every file was expected to rotate in a precise trajectory [Figure 2].

First, the endodontic micromotor and contra-angle handpiece (16:1, Endomate DT, NSK, Japan) were



Figure 1: Cryocan.

placed in the support arm in a parallel position with the apparatus base. Then, to ensure the correct locking, the file was meticulously secured to the contra-angle handpiece. Next, as instructed by the manufacturer, the electric motor was calibrated to run at a speed of 500 rpm and a torque of 2.5 N-cm. Accurate measurement as well as instrument placement could guarantee the file fixation. The micromotor was turned on as soon as the instrument was well positioned in the canal.

A tempered glass was used to cover the artificial canal to stabilize the instrument and to allow for observation of the rotating instrument.

The canal was lubricated using synthetic oil (white grease lubricant, Bison International, Goes, the Netherlands) to reduce the friction of the instruments.

Fracture testing time for each group was recorded with a digital stopwatch (Casio, Tokyo, Japan). This was a manually operated test, taking from the moment the motor went on to the moment a fracture was detected. This procedure was sequentially repeated by the same operator for all instruments in the both groups.

The time to fracture (TF) was multiplied by the number of rotations per minute to obtain the number of cycles to fracture (NCF) for each instrument.

Statistical analysis

Once all the tests had been administered, the mean values were calculated for each group.

Before data analysis, a Kolmogorov–Smirnov test was conducted ($P > 0.05$) to evaluate if the t -test in each group could meet the requirements of the

normal distribution. Independent-samples t -test of software (SPSS for Windows 22.0; SPSS, Chicago, IL, USA) was used to compare the control group with CT group at a statistical significance level of $P < 0.05$.

RESULTS

The Kolmogorov–Smirnov results indicated that the data were normally distributed.

Mean values of NCF and TF of each HyFlex[®] CM rotary endodontic instrument following rotation in artificial root canals with 60° angle of curvature was observed [Table 1]. T -test analysis revealed that although HyFlex[®] CM rotary file resistance to cyclic fracture was lower in cryogenically treated group than in the control group, the difference was not statistically significant ($P > 0.05$).

DISCUSSION

The fracture behavior of rotary instruments might be affected by the cold treatment processes that could lead to a potential source of changes to the crystalline alloy structure.^[12] Therefore, it goes almost without saying that it is necessary to examine the impact of surface treatment procedures on how NiTi rotary instruments perform.

In this study, the null hypothesis was confirmed and although CT could result in a reduction in the cycles of the fracture in the group receiving the process, there was no statistically significant difference in the number of cycles in control group. Therefore, CT had a limited impact on reducing the number of the cycles ending to fracture of Hyflex rotary file.

However, Gavini *et al.* examined the cyclic fatigue resistance of the K3 NiTi rotary instruments submitted to nitrogen ion implantation.^[11] The results showed that ion implantation was effective in improving the resistance of rotary NiTi instruments to the cyclic fatigue.

The present study differed in procedure with the one designed by Gavini *et al.*, in that they examined



Figure 2: A dental handpiece mounted on the block with the instrument inserted in an artificial root canal.

Table 1: Study results in different groups

| Category | <i>n</i> | Number of cycles to fail ^a | Time to fail (sec) ^a |
|-------------------------|----------|---------------------------------------|---------------------------------|
| Cryogenic instrument | 10 | 1395±350 | 167.4±42 |
| Noncryogenic instrument | 10 | 1576±236 | 189.1±28 |

^aIndependent-samples t -test, data are expressed as a mean±SD. SD: Standard deviation

how implantation of the nitrogen ion could affect the properties of the instruments. Implantation occurs within the near-surface layer (below 1 mm deep), and hence in this case, implantation becomes a source of compressive residual stress at the subsurface level, and it is able to inhibit the formation and propagation of fracture lines.^[11]

Yet, the study performed by George *et al.* indicated that the fracture time was significantly more in three different rotary files (Hero Shaper, RaCe, and K3) after deep dry CT.^[4]

George *et al.* concluded that the increase in the level of hardness could be attributed to the alloy completely transforming from the austenitic to martensitic phase. Such a change in phases normally occur at -195°C and could result in a reduction in the level of internal stress within the alloy due to the plastic deformation.^[4]

The study differed in findings with those of George *et al.*; this contrast may be attributed to the different CT procedure in each study as the latter applied the cryogenic cooling machine to allow the conditions of controlled dry thermal treatment. The procedure prevents from sample immersion in liquid nitrogen and eliminates the risk of thermal shock.^[2]

Another controversial topic may be due to the use of rotary instruments while Hyflex is made of cm wire making the files extremely more flexible than conventional NiTi rotary files.

The X-ray diffraction results revealed that no titanium nitride was formed following CT. They also suggested that contrary to ion implantation, CT leads nitrogen to deposit into the interstitial layers of the atomic lattice of the alloy.^[5]

According to the results of energy-dispersive X-ray spectroscopy line profile analysis, after CT nitrogen was evenly distributed throughout the entire cross-section of the instrument.^[5] The difference between the results of the present study and those of Gavini *et al.* could be explained by applying two different processes with different results in the way nitrogen is distributed.^[11]

It goes without saying that the instruments' mechanical behavior is highly influenced by the nature of the alloy and the manufacturing process. In addition, even slight changes in composition, impurities, and heat treatment conditions could have a considerable impact on mechanical properties of NiTi alloy.^[13] As a case in point, heat treatment (as a part of the manufacturing

process) strongly affects two properties of super elasticity and shape memory.^[13]

The present study examined the effect of CT cyclic fatigue resistance of rotary Hyflex instruments (Coltene-Whaledent, Altstetten, Switzerland). In this file, there is a lower percentage of nickel concentration (52 Ni %wt) than found in the majority of commercially available NiTi rotary instruments (54.5–57 Ni %wt).^[14]

The values are within the nominal composition range specified in the American Society for

Testing and Materials (ASTM) standards for wrought NiTi alloys that are commonly applied in medical devices and surgical implants (F2063-05 2005).^[15]

Shen *et al.* illustrated that NiTi instruments made of CM Wire were nearly 300%–800% more resistant to fatigue failure than those made of conventional NiTi wire.^[13]

In another study, Capar *et al.* recently found that HyFlex® files showed a higher fatigue resistance compared to ProTaper Next and Revo-S.^[16] Moreover, a large number of studies showed that NiTi rotary instruments produced by the new CM-wire manufacturing process enjoyed more flexibility as well as resistant to cyclic fatigue than those with a traditional manufacturing process or a thermally treated NiTi alloy called M-wire.^[10,13]

This could justify the increase in the mean NCF in the present study (1576 cycles in control group). This is in contrast with the similar studies like the research by Gavini *et al.*, in which the mean NCF in file K3 was recorded to be 381 cycles in the control group.^[11]

Although manufacturers have not publicized the information concerning the exact thermomechanical treatment of CM wires, the mechanical deformation behavior is closely attributed to the temperature of the phase transformation that is in turn reliant on the material's thermomechanical history.^[17]

The results of the present study suggest that the slight decrease in the mean of cycles ending to the fracture in HyFlex instruments probably resulted from (1) the proprietary manufacturing process (with proprietary heating and cooling processes) and/or (2) the difference in weight of nickel in Hyflex instruments, both of which might be inconsistent with CT.

It is complicated to determine that, property thermal proportion, unique composition, or both,

are responsible for improving the flexibility of the file. As reported by several studies, mechanical properties of NiTi instruments (including flexibility) are influenced by phase transformation behavior, and factors such as chemical composition, heat treatment, and manufacturing processes could easily impose an impact on the mechanical properties.^[15,18-20]

In addition, in their study, Zinelis *et al.* revealed that Hyflex compositional deviations did not considerably affect its mechanical properties, but the thermomechanical history must be considered as a significant factor on the final mechanical strength.^[14]

Thus, it can be concluded that CT could not increase the cyclic fatigue resistance of CM wire rotary instruments and it is partially inconsistent. The findings could be associated with the Hyflex instruments with special thermal processes, which is absent in the traditional instruments.

Although the change in the weight of nickel may be the result of the manufacturing changes in the raw material or thermal processes, the question still remains if nickel concentration of different percentages could play any significant role in Hyflex instruments.^[14]

This change could be considered as a result of the thermomechanical history that is unique to the alloy. Therefore, it would be very difficult to decide whether it is a single factor or more likely a combination of different correlated factors that play role in determining the final mechanical properties of Hyflex instruments.

It is noteworthy that the present study did not directly correlate with the clinical conditions because designing the cyclic fatigue tests are not able to simulate such conditions with all the details. Therefore, these tests are only useful for the comparisons between files. Attention must also be paid to the absence of similarities in clinical conditions that never allows the instruments to rotate in the same position for a long time.

CONCLUSION

This study concludes that deep wet CT for 24 h did not improve the cyclic fatigue resistance of HyFlex® CM rotary instruments.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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