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Mechanical behavior of deep cryogenically treated martensitic shape memory nickel–titanium rotary endodontic instruments

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ABSTRACT

Objectives: The aim of this study was to investigate the role of deep cryogenic treatment (DCT) on the cyclic fatigue resistance and cutting efficiency of martensitic shape memory (SM) nickel–titanium (NiTi) rotary endodontic instruments. **Materials and Methods:** Seventy-five HyFlex® CM instruments were randomly divided into three groups of 25 each and subjected to different DCT (–185°C) conditions based on soaking time: DCT 24 group: 24 h, DCT 6 group: 6 h, and control group. Each group was randomly subdivided for evaluation of cyclic fatigue resistance in custom-made artificial canals ($n = 15$) and cutting efficiency in plexiglass simulators ($n = 10$). The cyclic fatigue resistance was measured by calculating the number of cycles to failure (NCF) and cutting efficiency was measured using the loss of weight method. **Results:** Increase in NCF of instruments in DCT 24 group was highly significant ($P < 0.01$; Tukey's honest significant difference). There was no difference in weight loss of plexiglass simulators in all the groups ($P > 0.05$; one-way analysis of variance). In conclusion, deep dry cryogenic treatment with 24 h soaking time significantly increases the cyclic fatigue resistance without affecting the cutting efficiency of SM NiTi endodontic instruments.

Key words: Cutting efficiency, cyclic fatigue, deep cryogenic treatment, nickel–titanium, shape memory

INTRODUCTION

The rotary endodontic instruments manufactured from nickel–titanium (NiTi) alloy play an important role of preparing smooth tapered walls in curved root canals.[1] Although NiTi alloys possess the exotic properties of shape memory (SM) and superelasticity, instrument separation inside the canal without warning is a potential problem for the clinicians.[2] To overcome this difficulty, several thermomechanically processed NiTi instruments with stable martensite phase at body temperature were introduced.[3,4] These instruments possess the controlled SM with remarkable fatigue resistance up to 300% as claimed by the manufacturer.[5]

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Despite the extreme flexibility,[4] these thermomechanically processed NiTi instruments undergo permanent plastic deformation, especially while reusing the smaller size instruments[3,6] due to inadequate martensite content. Cryogenic treatment is a supplementary procedure of subjecting stainless steel[7,8] and superelastic NiTi[9] to sub-zero temperatures that affects the entire bulk of the material rather than the accessible surface alone. One of the mechanisms proposed behind the improvement in properties following cryogenic treatment is the complete martensitic transformation from the austenite phase.[10] It has been proved that deep cryogenic treatment (DCT) at a soaking temperature of up to −185°C increases the martensite content of SM NiTi alloys by a similar mechanism.[11]

An ideal rotary NiTi instrument should adequately possess the qualities of fracture resistance and faster cutting action.[12] Therefore, the optimal balance between the fatigue resistance and cutting efficiency of the instrument would result in a safe and quick preparation of the root canal. It is well known that instrument separation occurs either by brittle fracture (cyclic fatigue) or by ductile fracture (torsional fatigue).[2] Since the cyclically fatigued instruments undergo work hardening while rotating in a curved root canal for a prolonged period, they eventually fracture without any warning.[13,14] Therefore, the operator should be more cautious toward brittle fracture due to cyclic fatigue.

However, the effect of DCT on the mechanical behavior of SM NiTi rotary endodontic instruments has not been evaluated so far in the literature. Hence, the purpose of this study was to evaluate the effect of deep dry cryogenic treatment on the cyclic fatigue resistance and cutting efficiency of martensitic SM NiTi instruments. The null hypothesis tested was that there are no differences in cyclic fatigue resistance and cutting efficiency of SM NiTi instruments following DCT at 24 h and 6 h soaking time.

MATERIALS AND METHODS

The martensitic SM NiTi instruments made from controlled memory wire (HyFlex® CM; Coltene Whaledent Inc., Cuyahoga Falls, OH, USA) of size 25, 0.06 taper were selected for this study. A total of 75 instruments were randomly divided into three groups of 25 each as follows: DCT 24: Soaking temperature - −185°C, soaking time - 24 h; DCT 6: Soaking temperature - −185°C, soaking time - 6 h; and control: No treatment. The cooling rate of 1°C/min (3 h) and warming rate of 0.6°C/min (6 h) was kept constant for the experimental groups. The experimental setup for DCT used herein has already been described in the previous study [Figure 1].[9] Each set of instruments from DCT 24, DCT 6, and control groups following DCT were randomly allotted for evaluation of cyclic fatigue resistance (n = 15) and cutting efficiency (n = 10).

Cyclic fatigue resistance

The cyclic fatigue testing device used herein has been made as described in previous studies.[15,16] The device consists of a main frame that is connected to an electric handpiece using a mobile plastic support, and the artificial canals within a stainless steel block. The artificial canal with 30° angle of curvature and radius of curvature of 5 mm was manufactured with dimensions matching with that of an instrument (tip size 25, 0.06 taper). The artificial canal was covered with a tempered glass for instrument stability in the rotation and for direct visualization of the instruments during fracture.

The HyFlex® CM (Coltene Whaledent Inc., Cuyahoga Falls, OH, USA) of size 25, 0.06 taper instruments (15 per group) were placed inside the artificial canal till its full length and then rotated freely rotated under oil lubrication (Super Oil, Singer, Elizabethport, NJ, USA) at 500 rpm and 2.5 Ncm torque using a digital torque control meter(X-Smart, Dentsply Maillefer Instruments SA, Ballaigues, Switzerland). The time taken by each instrument to fracture was visually recorded with a digital stopwatch. The time to fracture was multiplied by the number of rotations per minute to obtain the

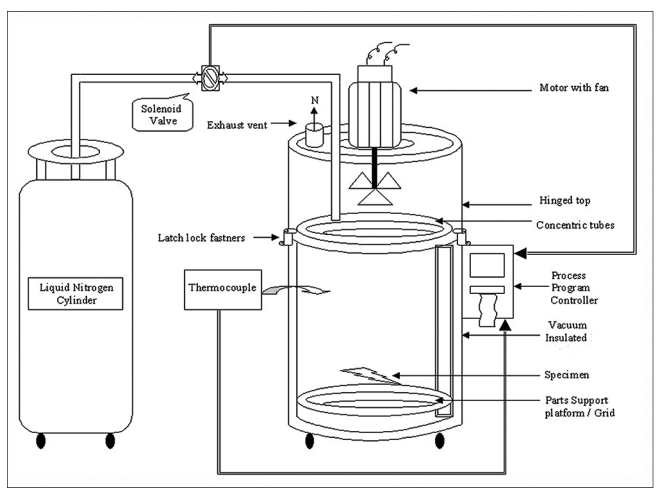

Figure 1: Experimental setup

number of cycles to failure (NCF) for each instrument. The NCF is directly proportional to the cyclic fatigue resistance of the instrument.

Cutting efficiency

New plexiglass simulators (Endo Training Bloc, Dentsply Maillefer Instruments SA, Ballaigues, Switzerland) with a standardized 16.5 mm long curved canal were used for the evaluation of cutting efficiency. The prestandardized canal was instrumented to the working length of 16.0 mm till size 20, 0.04 taper using Mtwo rotary endodontic instruments (VDW, Munich, Germany) using digital torque control motor (X-Smart; Dentsply Maillefer Instruments, SA) according to the manufacturer's instruction by crown-down technique. The canals were irrigated with 5 ml physiological saline solution. All HyFlex® CM (Coltene Whaledent Inc., Cuyahoga Falls, OH, USA) of size 25, 0.06 were introduced into resin blocks canal and rotated at a constant rate of 500 rpm and torque 2.5 Ncm. Each file was exposed to three sequences in the resin-blocks canals. Each cycle consisted of 16 subcycles of 5 s each for a total time of 80 s.[17] Copious irrigation with a physiological saline solution was carried out. The dried endodontic plexiglass simulators were weighed in an electronic weigh balance (Analytical Plus; Ohaus Corporation, Pine Brook, NJ, USA) before and after the preparation to calculate the loss of weight. The loss of weight of the plexiglass simulators is directly proportional to the cutting efficiency of the instrument.

Statistical analysis

The significance for NCF and loss of weight was analyzed using one-way analysis of variance (ANOVA) followed by *post hoc* multiple comparisons with Tukey's honest significant difference test by using SPSS software version 16.0 (SPSS Inc. Chicago, IL) at a 0.05 significance level.

RESULTS

Maximum NCF of each HyFlex® CM rotary endodontic instrument following rotation in artificial root canals with 30° angle of curvature was observed in DCT 24 group followed by DCT 6 and control. An ANOVA showed that the effect of DCT on the NCF was statistically significant between the groups ($P < 0.01$; one-way ANOVA). There was a significant ($P < 0.01$; Tukey's honest significant difference) increase in NCF for instruments in DCT 24 group when compared with that of DCT 6 and control [Table 1].

The loss of weight of each plexiglass simulators following instrumentation with HyFlex® CM rotary endodontic instrument is given in Table 1. Minimum loss of weight of observed in DCT 24 group. There was no difference in mean loss of weight of plexiglass simulators in among the groups ($P > 0.05$; one-way ANOVA).

DISCUSSION

Despite the extreme flexibility of HyFlex® CM SM NiTi instruments,[18] supplementary cryogenic treatment would further increase the volume of martensite[7] resulting in superadded benefits, especially for smaller size instruments. Cryogenic treatment can be classified based on the soaking temperature as deep (−185°C) and shallow (−80°C).[9,19,20] Deep soaking temperatures are effective in increasing the martensite content of SM NiTi alloys than the conventional shallow cold treatments.[11] Consequently, DCT significantly reduces the hardness and increases the wear resistance of the SM NiTi alloy.[21] Therefore, the objective of this study was only to evaluate the role of DCT soaking time with respect to the constant cooling and warming rate. The advantage of the dry DCT facility used in this study is to gradually increase or decrease the temperature in order to avoid thermal shock to the instrument that would make it brittle.[9]

Based on the characterization experiments performed earlier,[11,21] it is evident that DCT has a significant role in reducing the hardness of the Ni51Ti49 alloy thereby indirectly improving the flexibility[22] of the rotary instrument made out of this new alloy. Hence, the evaluation of cyclic fatigue resistance and cutting efficiency was performed as a confirmatory experiment using commercially available martensitic SM NiTi rotary endodontic instrument (HyFlex® CM (Coltene Whaledent Inc., Cuyahoga Falls, OH, USA) of approximating composition[23] and active A_f.[24] However, there is a concern as to whether the reduction in wear resistance[21] would compromise

Table 1: Intergroup comparison of number of cycles to failure (*n*=15) and weight loss values (*n*=10) of shape memory nickel-titanium instruments

	Number of cycles to failure†	Weight loss† (×10⁻³ g)
DCT 24	2280±205[a]	212±84[d]
DCT 6	2026±93[b]	215±94[d]
Control	2011±219[b]	236±78[d]

†One-way ANOVA and Tukey's *post hoc* test, data are expressed as a mean±SD, values with different superscript letters indicate a statistically significant difference ($P<0.05$). SD: Standard deviation, DCT: Deep cryogenic treatment, ANOVA: Analysis of variance

the cutting efficiency of martensitic SM NiTi rotary endodontic instruments and subsequently the chair side time of the patient.

The more commonly occurring torsional fracture of NiTi rotary instruments arises out of single overload incident, which may be caused by using too much apical force during instrumentation or very small preexisting canal size due to lack of preliminary hand instrumentation.[14] The operator can control these iatrogenic factors by taking necessary precautions. In contrast, the operator could not control the angle and radius of the root canal curvature that results in cyclic flexural fatigue, which in turn decreases the instrument lifespan *in vitro* and clinically.[14]

The main advantage of the cyclic fatigue testing device used in this study is that each artificial canal is customized for the dimensions of the instrument tested giving it a precise and reproducible trajectory.[14,15,25] Size 25, 0.06 taper was selected because it was the master apical NiTi file in many operative sequences. Schäfer *et al*.[26] had reported that root canals with <27° angle of curvature had 65% prevalence followed by those between 27° and 35° with a prevalence rate of 13%. Hence, an artificial canal with 30° angle of curvature was selected for this study. The severity of curvature for a given angle of curvature can be categorized based on the radius of curvature as severe (5 mm), moderate (7 mm,) and mild (10 mm).[27] A 5 mm radius of curvature was selected for this study in order to simulate the worst testing conditions prevailing in the oral cavity.

There is a drastic increase in cyclic fatigue resistance of HyFlex® CM instruments of by 13% and <1% in DCT 24 and DCT 6 groups, respectively when compared with the control group. HyFlex® CM instruments possess a mixture of austenite and martensite phase at room temperature.[28] The enhanced cyclic fatigue life of SM NiTi rotary instruments following DCT could be attributed to the presence of relatively more martensite phase[11] when compared with the nontreated instruments. Consequently, there is a reduction in hardness[21] and increase in flexibility of the SM NiTi alloy as these two properties are inversely proportional to each other.[22] A fully developed martensite phase at body temperature has a maximum recoverable strain of up to 9%.[29] There was an additional increase in cyclic fatigue resistance of SM NiTi rotary instruments by 12% following DCT with 24 h soaking period when compared with 6 h. This could be due to the adequate time available for the complete transformation of retained austenite to the martensite phase[30] as observed in the experiments performed earlier on SM NiTi alloy.[11,21]

Basically, the martensite phase of the NiTi alloy possesses a relatively lower Young's modulus (20–50 GPa) and yield strength (138 GPa) than the austenite phase (40–90 GPa and 379 MPa, respectively).[31] This indicates that the martensite requires lower stress for deformation, whereas the austenite requires higher stress as they possess better yield strength. In addition to high flexibility than austenite, the martensite reduces the risk of instrument fracture under high stress due to its plastic deformation.[32]

Although various methods have been used to measure the cutting efficiency, such as weight loss method,[17] microcomputed tomography,[12] and profilometry,[33] it appears that there is no single universally accepted method. The simplest and predictable method of evaluating cutting efficiency would be the weight loss of substrate after cutting for a given time.[17,34] The various factors that influence the cutting efficiency of endodontic instruments are the substrate (dentin or plexiglass), cross-sectional configuration, tip design, flute and blade design, hardness and sharpness of the blade (cutting edge), lubrication during cutting, wear resistance of the material with which the instrument is made, and method of use.[1] Hence, all these factors were standardized except for the hardness and wear resistance of the instrument, which is due to the impact of DCT.

Cutting efficiency of endodontic instruments was examined by operating them on acrylic blocks (plexiglass) and human dentin. Some studies discouraged testing with human teeth because of their variable hardness and water content.[35,36] The advantages of using acrylic include providing a standardized environment for testing of identical samples, without variations in hardness, thermal properties, and canal shape that may influence results; however, this material does not reflect the clinical conditions of use and can be used for comparison purposes.[1,34]

The frictional heat generated by HyFlex® CM SM NiTi instruments melts some resins[1] and therefore physiological saline was used as an irrigant. Morgental *et al*.[34] observed remarkably higher cutting efficiency of HyFlex® CM coronal flaring instruments when compared with BioRace, ProFile, and ProTaper in lateral cutting action. In contrast, there was a reduction in cutting efficiency of HyFlex® CM SM NiTi instruments that were subjected to both DCT 24 and DCT 6. However, their difference with the control group was not significant in spite of decreased wear resistance of SM NiTi alloy.[21] The probable reason could be the alloy's thermomechanical processing and the flutes configuration, which could store a greater amount of dentin or acrylic chips.[34]

The soaking time played a significant role in increasing the cyclic fatigue resistance of HyFlex® CM SM NiTi instruments. However, the soaking time did not influence the cutting efficiency of the same. Hence, the null hypothesis was partially rejected.

Under the given standardized experimental conditions, deep dry cryogenic treatment with 24 h soaking time significantly increases the cyclic fatigue resistance of SM NiTi endodontic instruments without compromising the cutting efficiency and subsequently the total preparation time. Further investigation is required to evaluate the impact of DCT on cutting efficiency of these instruments on natural substrates.

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Conflicts of interest

There are no conflicts of interest.

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