Evidence for Reduced Fatigue Resistance of Contemporary Rotary Instruments Exposed to Body Temperature

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Abstract

Introduction: The purpose of this study was to evaluate the effect of 2 different temperatures (20°C and 37°C) on the cyclic fatigue life of rotary instruments and correlate the results with martensitic transformation temperatures. Methods: Contemporary nickel-titanium rotary instruments (n = 20 each and tip size #25, including Hyflex CM [Coltene, Cuyahoga Falls, OH], TRUShape [Dentsply Tulsa Dental Specialties, Tulsa, OK], Vortex Blue [Dentsply Tulsa Dental Specialties], and ProTaper Universal [Dentsply Tulsa Dental Specialties]) were tested for cyclic fatigue at room temperature (20°C \pm 1°C) and at body temperature (37°C \pm 1°C). Instruments were rotated until fracture occurred in a simulated canal with an angle curvature of about 60° and a radius curvature of 3 mm; the center of the curvature was 4.5 mm from the instrument tip. The number of cycles to fracture was measured. Phase transformation temperatures for 2 instruments of each brand were analyzed by differential scanning calorimetry. Data were analyzed using the t test and 1-way analysis of variance with the significance level set at 0.05. Results: For the tested size and at 20°C, Hyflex CM showed the highest resistance to fracture; no significant difference was found between TRUShape and Vortex Blue, whereas Pro-Taper Universal showed the lowest resistance to fracture. At 37°C, resistance to fatigue fracture was significantly reduced, up to 85%, for the tested instruments (P < .001); at that temperature, Hyflex CM and Vortex Blue had similar and higher fatigue resistance compared with TRUShape and ProTaper Universal. Conclusions: Under the conditions of this study, using a novel testing design, immersion in water at simulated body temperature was associated with a marked decrease in the fatique life of all rotary instruments tested. (J Endod 2016; 1:1-6

Key Words

Body temperature, cyclic fatigue, martensitic transformation

The development of nickel-titanium (NiTi) endodontic instruments has continued in the last 2 decades mainly because of the alloy's superelastic property, which is associated with a reversible phase transformation called a martensitic transformation (1–3). Thermomechanical processing and alloying treatments have been used to improve the superelasticity and fatigue resistance of these NiTi instruments (2, 4).

However, although endodontic instruments have been improved, fracture during treatment continues to be a challenge for clinicians (5). Rotary instrument fracture can occur through cyclic fatigue, which accumulates while the instrument rotates within a curvature (6). Alternatively, an instrument can fracture because of torsional failure when the elastic limit of the material is exceeded (7).

Different parameters have been used to evaluate the fracture resistance of endodontic instruments, such as angle and radius of curvature (6), single or double curvature (8), rotary speed (6), and geometry of the simulated canal (9). Furthermore, in the absence of an established testing norm, several experimental designs have been created, such as 3 rigid stainless steel pins in a pegboard (10, 11), tempered steel rod and block assemblies (7, 12), and slots in steel form blocks (9).

These methodologies allowed comparisons among instruments tested within each experimental design, and it was found recently that more martensitic alloys are more fatigue resistant than austenitic instruments (8, 13, 14). However, these tests were all conducted at room temperature, taking care not to generate measurable heat, which is believed to potentially change NiTi properties (15, 16).

These studies have not considered the intracanal temperature during canal preparation. Only 2 studies in the literature report on intracanal temperatures *in vivo* (17, 18). However, both of these studies are related to irrigation during endodontic treatment and do not consider mechanical root canal preparation.

Newer alloys are believed to have transformation temperatures much higher than those of conventional austenitic materials used in previous generations of rotary instruments (19) and may in fact transform close to body temperature (2). Therefore, the aim of this study was to test fatigue limits of current, thermally treated, NiTi rotary instruments during immersion in water at body temperature.

Materials and Methods

Instruments

Selected sizes of NiTi rotary instruments Hyflex CM (size #25/.06; Coltene, Cuyahoga Falls, OH), TRUShape (size #25/.06v; Dentsply Tulsa Dental Specialties, Tulsa, OK), Vortex Blue (size 25/.06, Dentsply Tulsa Dental Specialties), and ProTaper Universal (F2, Dentsply Tulsa Dental Specialties) were tested for cyclic fatigue in a water

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TABLE 1. The Mean and Standard Deviation (SD) of Number of Cycles to Fracture (NCF) of Instruments in Water at 20°C and 37°C

Instrument*	Size	Temperature (°C)	Mean	SD	Air
Protaper Universal	F2	20	199	41	126
		37	134	39	
Hyflex CM	25.06	20	2986	412	997
•		37	487	96	
TRUShape	25.06v	20	1372	335	408
		37	201	35	
Vortex Blue	25.06	20	1816	1176	523
		37	479	139	

All instruments tested had significant reduction of fatigue resistance at 37° C compared with room temperature (P < .001).

bath both at room temperature ($20^{\circ}\text{C} \pm 1^{\circ}\text{C}$) and at body temperature ($37^{\circ}\text{C} \pm 1^{\circ}\text{C}$). A subset of instruments was also tested in air, without immersion in water (Table 1).

Twenty instruments of each brand (tip size #25) were used in this study in each subgroup. No torque limit was applied; revolutions per minute (rpm) used were set according to the respective manufacturer guidelines (500 rpm for Hyflex CM and Vortex Blue and 300 rpm for ProTaper Universal and TRUShape).

Differential Scanning Calorimetry Analysis

Two instruments of each brand were used for differential scanning calorimetry (DSC) analysis; to prepare the samples for analysis, multiple sections of $1{\text -}4$ mm were cut from each instrument using manual diagonal cutting and weighed to an accuracy of ± 0.01 mg before being placed in a preweighed Tzero aluminum pan (TA Instruments, New Caste, DE) and nonhermetically sealed. Samples were prepared starting at the instrument tip in short sections with the aim of achieving a total sample weight of approximately $10{\text -}20$ mg.

Each sample was placed in a Q2000 DSC instrument (TA Instruments) along with an empty Tzero aluminum reference pan. The purge gas used was nitrogen at a flow rate of 50 mL/min. The samples were first heated to 60°C and then cooled to -60°C at a rate of $-10^{\circ}\text{C}/\text{min}$ followed immediately by a heating cycle at $10^{\circ}\text{C}/\text{min}$ up to 60°C . The heating/cooling cycle was performed 3 times per sample. Some samples were cycled at $\pm 90^{\circ}\text{C}$ instead because their transformations occurred at more extreme temperatures.

All data were analyzed using TA Instruments' Universal Analysis software. The starting and finishing temperatures were determined as the intersection of the line tangent to the curve at its point of inflection and the baseline. Baselines were selected using TA Instruments' sigmoid tangent method (selecting 4 points).

Cyclic Fatigue Analysis

A plastic base with 3 adjustable stainless steel pins was used to simulate the curvature of the root canal. Each pin was 6 mm in diameter and 4-cm long and had an approximately 0.5-mm-wide V-shaped notch in which instruments were positioned for the test. A 1:8 gear reduction handpiece connected to an electric motor (DT Endo, Dentsply Tulsa Dental Specialties), which allowed the selection of the number of revolutions (rpm) and torque limit, was attached to the same plastic base. The motor was connected to a microcomputer (Hawkins Electronics, San Rafael, CA) with a lead connected to 2 pins at the plastic base. The start button of the microcomputer started the motor and an electronic stopwatch (accuracy ± 0.1 seconds).

The design was such that, when the instrument fractured during the cyclic fatigue test, the electric circuit linked between the pins, motor,

and microcomputer was interrupted, stopping the time and determining the fatigue life after each instrument fractured (Fig. 1A and B). A pilot trial (n = 3 instruments per group) was performed with the plastic base placed in the empty water container, rotating the instruments until fracture in ambient air. Then, to be able to define temperatures, a glass container was filled with 200 mL deionized water at 20°C or 37°C. Deionized water was acquired from a Milli Q Integral unit (Millipore, Billerica, MA), and the original temperature of the water was 25.5°C. To achieve the desired temperatures, the glass container was either immersed in ice until the water temperature was stabilized at 20°C or placed on a hot plate until the water temperature was stabilized at 37°C; during all tests, the temperature was measured with an infrared thermometer. The plastic base with pins, the instrument, and 2 connected clips was positioned inside the glass container and fixed with a clamp, and sufficient time after immersion of the base was allowed to equilibrate temperatures.

All instruments were tested in a 3-mm radius of curvature with an angle of curvature of 60° . The center of the curvature was 4.5 mm from the tip, and the working length was 19 mm for each instrument tested (Fig. 1C). The radius and angle of curvature were determined according to Pruett et al (6). Cyclic fatigue times were recalculated into number of cycles until failure (NCF) to account for the different rpm settings. Because data were found to follow a normal distribution, NCF comparisons between temperatures were made by t tests, and NCFs within temperatures were compared using 1-way analysis of variance and Scheffe post hoc tests.

Results

DSC

All instruments were found to have homogeneous thermal transformations as a function of sample axial coordinates and were seen to be thermally stable (displayed repeatable DSC scans) over several cycles. However, the different instruments displayed a wide range of behaviors with varying transformation temperatures and different solid-solid phase transformations (Table 2). Some presented classic austenite to martensite transformation, presumably cubic to monoclinic, and the reverse. However, others included apparent intermediate phases, presumably R-phase (rhombohedral) structures.

ProTaper Universal displayed a classic austenite-martensite phase transformation centered close to 0°C (Fig. 2A); TRUShape showed classic R-phase transformations. However, during both the cooling and heating cycles, the R-phase and the martensitic transformations overlapped and could not be separated in terms of transformation temperature (Fig. 2B). Hyflex CM displayed classic R-phase transformations. Upon cooling, the austenite transformed to R-phase just below 20°C. There was a wide R-phase to martensite transformation (presumably monoclinic) well below 0°C. During heating, the martensite to Rphase and the R-phase to austenite transformations overlapped and could not be separated (Fig. 2C). Vortex Blue also showed classic R-phase transformations. Upon cooling, an austenite to R-phase transformation occurred at roughly 30°C. An R-phase to martensite transformation (presumable monoclinic) took place upon further cooling around -60°C. Upon heating, the martensite to R-phase transformation around 20°C precedes the R-phase to austenite transformation, which takes place around 30°C (Fig. 2D). In all plots, the cooling curve lies above the heating curve.

Cyclic Fatigue Resistance

The mean NCF and standard deviations for ProTaper Universal, Hyflex CM, TRUShape, and Vortex Blue at ambient temperature (20° C) and body temperature (37° C) are shown in Table 1. Fatigue resistance

^{*}N = 20 in all groups. Mean data from a pilot trial in air (n = 3 per group) are also given.





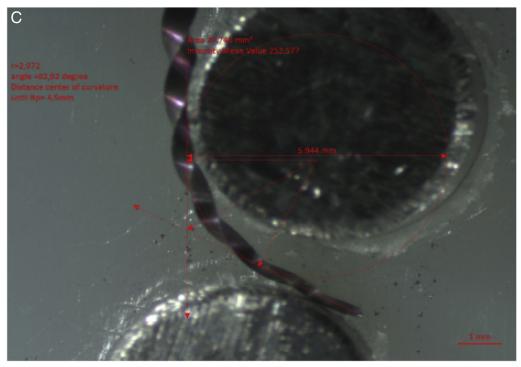


Figure 1. Experimental design with (*A*) the arrangement of the water bath (fluid level indicted with blue dye) and the rotary with pegboard arrangement and motor placed on the heating unit. The control unit is separate and has a digital readout. (*B*) The pegs are stainless steel rods with about a 6-mm diameter with a 0.5-mm notch. The selected geometry of the steel pins is shown in *C*, with indications of relevant measurements.

was significantly higher at ambient temperature compared with at 37° C, with differences ranging from 33%–85% (P < .0001, Table 1).

Fatigue resistance among instruments at each temperatures was also different; at 20° C, Hyflex CM had a significantly higher NCF than ProTaper Universal, TRUShape, and Vortex Blue (P < .0001, 1-way analysis of variance), whereas no significant difference was found between TRUShape and Vortex Blue (P = .2019); ProTaper Universal had a significantly lower NCF than Hyflex CM, TRUShape, and Vortex Blue (P < .0001). At body temperature, there was no significant difference between Hyflex CM and Vortex Blue NCFs (P = .9943), whereas both had significantly higher NCFs than ProTaper Universal and

TRUShape (P < .0001). No significant difference in NCFs was found between ProTaper Universal and TRUShape (P = .1306).

Discussion

This study aimed to compare fatigue life at ambient and body temperatures and relate the findings to martensitic/austenitic transformation temperatures. Several previous studies compared fatigue resistance between endodontic instruments under different environments (15, 19), including air and in a water bath (10). However, in vitro fatigue testing provides a simplistic analog to the clinical condition

Basic Research—Technology

TABLE 2. Transformation Temperatures and Associated Energy for Nickel-Titanium Rotary Instrument (mean \pm standard deviation for 3 samples of each instrument)

	Cooling			Heating			
Instruments	M _s (°C)	M _f (°C)	Q (J g ⁻¹)	A _s (°C)	A _f (°C)	Q (J/g)	
ProTaper Universal	9.2 (±0.2)	-7.7 (±1.1)	1.10 (±0.03)	-4.6 (±0.3)	10.5 (±0.7)	1.4 (±0.3)	
Hyflex CM TRUShape	−5 (±5) 30 (±4)	−32.3 (±1.3) 20 (±4)	5.6 (±1.4) 2.8 (±0.2)	21.5 (±1.7) 22 (±4)	43.5 (±0.7) 31 (±4)	14.4 (±0.4) 2.49 (±0.15)	
Vortex Blue				30.81 (0.08)	33.71 (±0.13)	2.25 (±0.15)	

A_b, austenitic finish temperature; A_s, austenitic start temperature; M_b martensitic finish temperature; M_s, martensitic start temperature; Q, latent heat of transformation.

(eg, selected specific canal curvatures are used). The current methodology is also limited (eg, only a single curvature was used).

Another limitation of previous studies was that ambient temperature has been used in previous fatigue experiments. Conversely, the methodology used in the present study allowed the evaluation of resistance to fracture of NiTi endodontic instruments under different temperature conditions. Importantly, it has been shown that *in vivo* intracanal temperature ranges from 31°C–35°C (17, 18); moreover, *in vitro* data suggest that irrigation solutions intracanal will be at body temperature within 30 to 60 seconds after deposition (20). These findings are in line with our pilot data using an infrared thermometer

indicating that rotary instruments just after removal from a root canal had surface temperatures of 30.8°C-32.5°C.

In agreement with previous findings (16, 21) the present data indicate that fatigue life in air is shorter than in water at room temperature, perhaps because of locally elevated temperatures (15, 16). To the best of our knowledge, the present study is the first to simulate cyclic fatigue test resistance for endodontic instruments under temperature conditions similar to those of the body.

Comparing the instruments tested at ambient temperature, Hyflex CM had a significantly higher fracture resistance followed by Vortex Blue and TRUShape, whereas ProTaper Universal showed a lower fracture

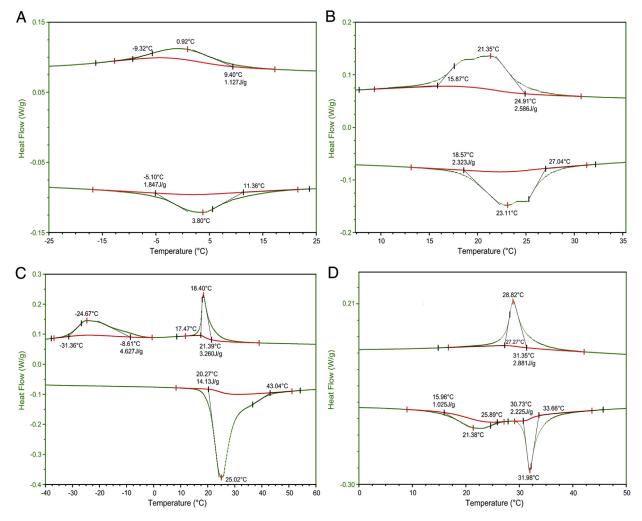


Figure 2. DSC curves indicating energy transfer as heat flow during cooling (upper trace) and heating (lower trace) of NiTi specimens. (*A*) ProTaper Universal, (*B*) TRUShape, (*C*) Hyflex CM, and (*D*) Vortex Blue.

Basic Research—Technology

resistance. These data support the belief that the more martensitic an NiTi alloy is the more flexible and hence the more fatigue resistant an instrument becomes (22). In fact, instruments with M-Wire (Sportswire LLC, Langley, OK) may prove more flexible and fatigue resistant than instruments manufactured with conventional NiTi wire (15), likely because of thermal processing in the creation of the alloy that produces a better arrangement of the crystal structure and changes in the relative percentage of phases present in the alloy (23).

Another possible reason for fatigue resistance is that the martensitic phase transformation has damping characteristics (2), which render crack propagation more difficult because of the larger number of interfaces present. A complex array of secondary cracks is formed because of these interfaces, dissipating the energy required for crack propagation (15).

At body temperature, fatigue resistance was found to be decreased for all instruments, however, to a different extent. To explain the significant reduction in fatigue life at room temperature, one may consider the pertinent transformation temperatures. DSC was used to analyze the phase transformation of NiTi endodontic instruments. In this analysis, certain peaks related to cooling and heating cycles indicate the martensitic (cooling) and austenitic (heating) transformations (1, 19, 24).

Specifically, we found austenitic finish (A_f) temperatures ranging from about 44°C (HyflexCM) to 10°C (ProTaper Universal), with TRUShape and Vortex Blue just below body temperature. This would suggest that ProTaper Universal is in an austenitic state at both temperatures tested here, whereas the 3 others are in varying degrees of martensitic condition at room temperature and move to a much more austenitic state at body temperature. The specific peak shape for Hyflex CM suggests that the conversion is not fully finished at body temperature but that a significant proportion is already austenitic.

The literature supports the finding that conventional NiTi is completely austenitic at oral temperature, displaying a higher modulus of elasticity (22); here, it was found that the $A_{\rm f}$ temperature of Hyflex CM was about 44° C, suggesting that this instrument at body temperature will be in a mixed martensitic R-phase and austenitic structure. Only limited information is available for DSC curves for Vortex Blue and TRUShape; recently published findings (25) as well as our data suggest a higher degree of austenite for Vortex Blue at body temperature compared with Hyflex CM. In contrast, ProTaper Universal has highest degree of austenite at body temperature.

Data from present DSC measurements suggested some explanations for the decrease of fatigue resistance at body temperature for heat-treated instruments. Hyflex CM, Vortex Blue, and TRUShape displayed classic R-phase transformations and higher $A_{\rm f}$ values. However, the crystal lattice of ProTaper Universal is likely very similar at room and body temperature; yet, fatigue resistance is reduced, albeit to a lesser extent. This suggests that other yet unknown factors may play a role in reducing fatigue resistance below previously documented values. Additional experiments are needed to address this question. The design for future fatigue tests should consider the simulation of environmental conditions such as temperature.

Conclusion

Under the limitations of this study, the following may be concluded:

- The methodology used in this study allowed the simulation of different temperatures during cyclic fatigue tests, including body temperature.
- A temperature increase to 37°C, simulating body temperature, substantially decreased the fracture resistance of all instruments tested.
- 3. Future fatigue tests should include the simulation of environmental conditions such as temperature.

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The remaining authors deny any conflicts of interest related to this study.

References

- Thompson SA. An overview of nickel-titanium alloys used in dentistry. Int Endod J 2000;33:297–310.
- Shen Y, Zhou HM, Zheng YF, et al. Current challenges and concepts of the thermomechanical treatment of nickel-titanium instruments. J Endod 2013; 39:163–72.
- Wayman CM, Duerig TW. An introduction to martensite and shape memory. In: Duerig TW, Melton KN, Stöckel D, Wayman CM, eds. Engineering Aspects of Shape Memory Alloys. London: Butterworth-Heineman; 1990:3–20.
- Pereira ES, Peixoto IF, Viana AC, et al. Physical and mechanical properties of a thermomechanically treated NiTi wire used in the manufacture of rotary endodontic instruments. Int Endod J 2012;45:469–74.
- McGuigan MB, Louca C, Duncan HF. Clinical decision-making after endodontic instrument fracture. Br Dent J 2013;214:395

 –400.
- Pruett JP, Clement DJ, Carnes DL Jr. Cyclic fatigue testing of nickel-titanium endodontic instruments. J Endod 1997;23:77–85.
- Peters OA, Barbakow F. Dynamic torque and apical forces of ProFile.04 rotary instruments during preparation of curved canals. Int Endod J 2002;35: 379–89.
- Duke F, Shen Y, Zhou H, et al. Cyclic fatigue of ProFile Vortex and Vortex Blue nickel-titanium files in single and double curvatures. J Endod 2015;41: 1686–90.
- Perez-Higueras JJ, Arias A, de la Macorra JC, Peters OA. Differences in cyclic fatigue resistance between ProTaper Next and ProTaper Universal instruments at different levels. J Endod 2014;40:1477–81.
- Cheung GS, Darvell BW. Fatigue testing of a NiTi rotary instrument. Part 1: strain-life relationship. Int Endod J 2007;40:612–8.
- Shen Y, Qian W, Abtin H, et al. Effect of environment on fatigue failure of controlled memory wire nickel-titanium rotary instruments. J Endod 2012; 38:376–80.
- Kim HC, Yum J, Hur B, Cheung GS. Cyclic fatigue and fracture characteristics of ground and twisted nickel-titanium rotary files. J Endod 2010;36: 147–52.
- Plotino G, Grande NM, Cotti E, et al. Blue treatment enhances cyclic fatigue resistance of vortex nickel-titanium rotary files. J Endod 2014;40:1451–3.
- 14. Nguyen HH, Fong H, Paranjpe A, et al. Evaluation of the resistance to cyclic fatigue among ProTaper Next, ProTaper Universal, and Vortex Blue rotary instruments. J Endod 2014;40:1190–3.
- Pereira ES, Gomes RO, Leroy AM, et al. Mechanical behavior of M-Wire and conventional NiTi wire used to manufacture rotary endodontic instruments. Dent Mater 2013;29:e318–24.
- Tobushi H, Hachisuka T, Yamada S, Lin PH. Rotating-bending fatigue of TiNi shapememory alloy wire. Mech Mater 1997;26:35

 42.
- Cunningham WT, Balekjian AY. Effect of temperature on collagen-dissolving ability
 of sodium hypochlorite endodontic irrigant. Oral Surg Oral Med Oral Pathol 1980;
 49:175–7.
- de Hemptinne F, Slaus G, Vandendael M, et al. In vivo intracanal temperature evolution during endodontic treatment after the injection of room temperature or preheated sodium hypochlorite. J Endod 2015;41:1112–5.
- Miyai K, Ebihara A, Hayashi Y, et al. Influence of phase transformation on the torsional and bending properties of nickel-titanium rotary endodontic instruments. Int Endod J 2006;39:119–26.
- Zeltner M, Peters OA, Paque F. Temperature changes during ultrasonic irrigation with different inserts and modes of activation. J Endod 2009;35:573

 –7.
- Shen Y, Zhou H, Campbell L, et al. Fatigue and nanomechanical properties of K3XF nickel-titanium instruments. Int Endod J 2014;47:1160–7.

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- Santoro M, Nicolay OF, Cangialosi TJ. Pseudoelasticity and thermoelasticity of nickeltitanium alloys: a clinically oriented review. Part I: temperature transitional ranges. Am J Orthod Dentofacial Orthop 2001;119:587–93.
- Shen Y, Coil JM, Zhou H, et al. HyFlex nickel-titanium rotary instruments after clinical use: metallurgical properties. Int Endod J 2013;46:720–9.
- Alapati SB, Brantley WA, Iijima M, et al. MIcro-XRD and temperature-modulated DSC investigation of nickel-titanium rotary endodontic instruments. Dent Mater 2009;25: 1221–9.
- 25. Shen Y, Zhou H, Coil JM, et al. ProFile Vortex and Vortex Blue nickel-titanium rotary instruments after clinical use. J Endod 2015;41:937–42.

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de Vasconcelos et al.