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ORIGINAL ARTICLE



Variable impact by ambient temperature on fatigue resistance of heat-treated nickel titanium instruments

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Abstract

Objectives The purpose of this study was to evaluate the effect of different ambient temperatures on cyclic fatigue (CF) life of two NiTi rotary systems and correlate the results with martensitic transformation temperatures.

Materials and methods Heat-treated NiTi Vortex Blue (VB) and EdgeSequel Sapphire (SP) instruments (tip sizes no. 20, 25, 30, 35, 40) were tested for CF resistance at room and body temperature (n = 20 each group) in a simulated canal (angle of curvature 60°; radius 3 mm; center from instrument tip 4.5 mm) with a motor controlled by an electric circuit. Mean half-life, beta and eta Weibull parameters were determined and compared. Two further instruments of each brand were subjected to differential scanning calorimetry (DSC).

Results Temperature had an effect on fatigue behavior: all instruments lasted significantly longer at room than at body temperature. All VB significantly outlasted those of SP at body temperature; while smaller diameters of VB (size no. 20) were also significantly more resistant than SP when tested at room temperature; SP with larger diameters (sizes no. 30, no. 35, and no. 40) lasted significantly longer than VB did.

Conclusions Immersion in water at body temperature was associated with a marked decrease in the fatigue life of all rotary instruments tested. VB instruments were significantly more CF resistant at body temperature and showed the highest predictability in terms of fracture resistance.

Clinical relevance Rotary instruments manufactured with different post-machining heat treatment responded differently to changed ambient temperatures. DSC assessment of martensitic conversion temperatures helps to predict the behavior of nickel titanium rotaries in different environments.

Keywords Body temperature · NiTi · Cyclic fatigue · Martensitic transformation temperature

Introduction

Walia et al. [1] first described endodontic instruments made from nickel titanium (NiTi) alloy; the physical properties of

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these files subsequently allowed an improvement of cleaning and shaping procedures [2]. At the same time, the increased use of NiTi files appeared to be associated with increased risk of intracanal instrument fracture, reported to be 0-9% [3] up to 21% [4] during endodontic treatment.

Clinical outcome studies suggested no significant change in success rates for roots with retained fractured instrument compared to controls, at least in the hands of experienced operators [3]. However, a retained instrument fragment will prevent proper cleaning and disinfection of the canal, which might affect the outcome of cases with preoperative periapical periodontitis [3].

Studies in controlled in vitro environments demonstrated that fractures are due to either cyclic fatigue (CF) or torsional failure [4]; although, it is likely that a combination of both factors contributes to the failure of NiTi instruments in clinical conditions [5]. In fact, numerical simulation has demonstrated that the cross-sectional design of the instrument plays an

important role on the stresses developed in the instrument under either torsion or bending [6].

Torsional failure occurs when a part of the instrument, usually the tip, is binding to the canal walls while the file is still rotating. When the stress state within the instrument exceeds the elastic limit of the metal, instruments will undergo plastic deformation and ultimately fracture [4]. Therefore, torsional failure may be prevented by the appropriate use of the files after confirming presence of a glide path and avoiding undue apical forces. In addition, the deformation, if the file does not fracture immediately, may be considered as a warning sign if the practitioner inspects the file regularly. On the other hand, CF accumulates when the file is rotating within a curvature, fracture typically occurring without any warning [7].

Multiple studies have evaluated the CF resistance of endodontic instruments. In the current absence of a testing norm, a variety of methods and designs has been used with different variables such as angle and radius of the curvature, or even single or double curvatures. In general, instruments tend to be less fatigue resistant in severe curvatures [8] and the radius of curvature was found to be the most significant factor effecting fatigue resistance of files [7]. Until recently, CF studies were performed in air and at room temperature. More recent studies [8–11] tested files in water baths and at body temperature and consistently found a significant decrease in CF resistance when tested in body temperature compared to ambient temperatures.

EdgeEndo is a manufacturer that has emerged in recent years with a variety of heat-treated NiTi rotary instruments that are claimed to have unmatched physical properties [12]. Among all the instruments marketed, EdgeSequel Sapphire was designed to be used exactly like Vortex Blue instruments (Fig. 1) but was claimed to be two times more fatigue resistant [12]. However, these instruments have not yet been independently tested under standardized fatigue conditions at both room and body temperature yet.

Therefore, the aim of this study was to compare CF resistance of EdgeSequel Sapphire in comparison to Vortex Blue. Moreover, the martensitic transformation temperatures for both instruments were determined using differential scanning calorimetry.

The null hypothesis of this study was that, across different instrument sizes, EdgeSequel Sapphire and Vortex Blue are similarly affected by a change from room to body temperature. A secondary hypothesis was that the behavior is matched by similar martensitic transformation temperatures.

Materials and methods

Instruments and settings

The two types of NiTi instruments tested in this study were EdgeSequel Sapphire (SP) (EdgeEndo, Albuquerque, NM, USA) and Vortex Blue (VB) (Dentsply Sirona, York, PA, USA) (n = 40 for each size no. 20, no. 25, no. 30, no. 35, and no. 40, all in 0.04 taper) giving a total of 400 instruments tested. Twenty instruments of each file type and size were tested in a water bath at two conditions, room (21 ± 1 °C) and body temperature (37 ± 1 °C).



Fig. 1 Scanning electron micrographs of Vortex Blue (a) and Edge Sequel Sapphire (b) size no. 25.04 rotary instruments, demonstrating visibly similar design patterns. Original magnifications are \times 15, \times 25, and \times 50, left to right

Both instruments tested have a constant 0.04 taper, triangular cross-sections, and similar lateral appearance (Fig. 1). A Pro-Mark motor and hand piece with 1:8 gear reduction (Dentsply Sirona) was used with no torque limit. EdgeEndo offers SP as compatible with VB and other 0.04/0.06 taper rotary file systems [12] and to be used in the same hand piece with the same speed and torque. A rotational speed of 500 rpm is recommended for SP [13]. The same rotational speed is recommended for VB [14]; therefore, the motor settings were identical for all instruments tested in the present study.

Cyclic fatigue testing

Three stainless steel pins were manufactured and coated with nitride with a top layer of ZrN (Surface Solutions, Fridley, MN, USA) to ensure appropriate corrosion and wear resistance. Each pin was 6 mm in diameter, 4 cm long, and had a 0.5-mm-wide V-shaped notch that allowed positioning the instruments for the fatigue test (Fig. 2). Pins and hand piece were attached to a pegboard base to simulate a canal with 3mm radius and 60° angle of curvature. The center of the curvature was 4.5 mm from the tip, and the hand piece was placed in a reproducible position for each instrument tested. The radius and angle of curvature were determined according to Pruett et al. [7]. A microcomputer (Hawkins Electronics, San Rafael, CA, USA) lead was connected to 2 pins at the plastic base and to the motor. The design was set so that an intact instrument electrically connected the two pins and completed the circuit, the microcomputer, and the stopwatch (accuracy ± 0.1 s). On manual initiation, the microcomputer started the motor and stopwatch, when the instrument fractured the circuit opened, which in turn caused motor and timer to stop.

The pegboard base with pins and the instrument were prepositioned inside a glass container filled with 200 ml of deionized water acquired from a MilliQ Integral unit (Millipore, Billerica, MA, USA) and fixed with a clamp. A precision mercury glass laboratory thermometer was also attached to the glass container to determine the temperature during the complete test. The first set of tests was performed at room temperature (21 °C). If temperature raised from 21 °C, ice was added until temperature was stabilized. The second set of tests was performed at body temperature (37 °C). To achieve body temperatures, the glass container was placed on a hot plate and the water temperature was stabilized [11].

DSC analysis

Two instruments of each brand were tested using differential scanning calorimetry (DSC) analysis (with scans ranging from approximately + 90 to -60 °C) to assess transformation temperatures and phase transformations. Sample preparation consisted of manual diagonal cutting of each instrument in short sections [length = 1 to 4 mm/weight =10-20 mg] starting at the tip. Sections were weighed to an accuracy of ± 0.01 mg before being placed in a pre-weighed Tzero aluminum pan (TA Instruments, New Castle, DE, USA). Each sample was then placed in a Q2000 DSC instrument (TA Instruments) along with an empty Tzero aluminum reference pan. Nitrogen at a flow rate of 50 ml/min was used as the purge gas. The samples were first heated to 90 °C and then cooled to -60 °C at a rate of -10 °C/min followed immediately by a heating cycle at 10 °C/min up to 90 °C. The heating cooling cycle was performed three times per sample. All data was 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' sigmoidal tangent method.



Fig. 2 Cyclic fatigue platform. a Arrangement of metal pins with surface coating. b Arrangement of the water container and thermometer placed on the heating device and the hand piece mounted to the pegboard

Statistical analysis

In additional to descriptive statistics, fatigue life spans were compared using Weibull analysis (Weibull ++7; Reliasoft Corporation, Tucson, AZ, USA). Weibull parameters and their 95% confidence intervals were calculated for each group as follows (see Fig. 3):

- 1. Mean life (seconds): the expected or average time to failure
- 2. Beta: the slope or shape parameter (dimensionless), the values of which are equal to the slopes of the regressed lines in the Weibull probability plot and are particularly significant because they provide a clue to the physics of the failure.
- 3. Eta (seconds): characteristic life or scale parameter. Eta is the typical time to failure in Weibull analysis and is related to the mean time to failure and defined as the expected time that 63.2% of the files will attain without breakage [15].

Results

Cyclic fatigue

Mean life, eta, and beta parameters and their 95% confidence intervals are shown in Table 1. All instruments lasted

significantly longer when tested at room temperature (21 $^{\circ}$ C) compared to body temperature (37 $^{\circ}$ C).

Temperature had an important and differential effect on fatigue behavior when comparing VB and SP instruments. Fatigue resistance varied for SP and VB instruments regarding the different tested diameters and temperatures. All VB instruments were significantly more resistant to CF than SP at body temperature. However, at room temperature, smaller diameters of VB (no. 20, 25) behaved differently from SP larger diameters (no. 30, no. 35, and no. 40) for which SP was more fatigue resistant.

Specifically, for size no. 20, VB instruments significantly outlasted those from SP both at room and body temperature with a probability of respectively 68 and 64%. For the rest of the sizes, no. 25, no. 30, no. 35, and no. 40, when tested at body temperature, VB significantly outlasted those of SP with probabilities of 97, 93, 67, and 84%, respectively. Conversely, when tested at room temperature, instruments no. 30, no. 35, and no. 40 of SP rotary system lasted significantly longer than corresponding VB instruments with probabilities of 70, 97, and 98%, respectively. There were no significant differences for instruments size no. 25 when testing VB and SP files at room temperature.

At the same time, larger beta parameters (Table 1) were calculated for VB instruments in general when compared to SP both at body and room temperature suggesting a higher predictability. The slope of the line in the Fig. 3 also confirmed this observation.



Fig. 3 Weibull probability plots [unreliability, CF (time) in Y axis versus time (s) in X axis] per group for sizes no. 20, no. 25, no. 30, no. 35, and no. 40

	Instrument	Temperature (°C)	Mean life (CI 95%)			Beta β (CI 95%)			Eta n (CI 95%)		
System			Mean life	(CI 95%)		Beta β	(CI 95%)		Eta η	(CI 95%)	
				Lower	Upper		Lower	Upper		Lower	Upper
SP	20	21	252.5	212	300.7	3.1	2.3	4.2	282.3	242	329.2
		37	82.9	71.1	96.8	3.3	2.3	4.7	92.4	80.4	106.2
	25	21	340.8	289.1	401.8	3.4	2.4	4.6	379.6	328.9	438
		37	79.2	69.4	90.3	4	2.39	5.6	87.3	77.8	98
	30	21	264.2	226.4	308.2	3.4	2.5	4.7	293.9	256.4	336.8
		37	51.2	44	59.7	4.1	3	5.4	56.5	49.5	64.4
	35	21	328.5	306.7	352	7.6	5.3	10.7	349.8	329.2	371.8
		37	77.1	67.7	87.9	5	3.8	6.6	84	75.2	93.9
	40	21	246.8	224.8	270.9	7	5.1	9.5	263.9	244.2	285.1
		37	56.5	49	65.3	3.7	2.7	5.2	62.6	55.2	71
VB	20	21	311.7	270.7	359	3.8	2.8	5.4	344.7	305	389.6
		37	93.9	88.3	99.9	9.5	6.7	13.4	99	94.1	104.1
	25	21	341.7	311.7	374.7	5.5	3.8	8	370.1	340.3	402.5
		37	141.4	130.9	152.7	7.4	5.4	10.1	150.7	141.2	160.8
	30	21	214.4	200.9	228.7	8.6	6.1	12	226.9	214.8	239.6
		37	97.7	86.5	110.3	4.1	2.8	5.9	107.6	95.9	120.7
	35	21	147.1	133.2	162.5	6.3	4.7	8.3	158.2	145.4	172.1
		37	87.5	81	94.5	6.7	4.7	9.7	93.7	87.5	100.3
	40	21	126.4	110.2	144.9	3.7	2.6	5.3	140	123.7	158.4
		37	80.8	73.5	88.8	5.5	3.9	8	87.5	80.5	95

 Table 1
 Mean life, eta, and beta parameters and their 95% confidence intervals

All instruments were significantly more resistant at room temperature. All VB instruments were significantly more resistant to CF than SP at body temperature

DSC analysis

The martensitic transformation temperatures determined by DSC analysis for SP and VB files are shown in Fig. 4. SP experienced a distinctly separated two-stage phase transformation. Upon cooling, it first displayed a classic R-phase transformation, followed by a transformation to an apparent monoclinic phase. Upon cooling, the transformation from austenite to R-phase starts around 35 $^{\circ}$ C and finishes at 15 $^{\circ}$ C and the transformation from



Fig. 4 DSC transformation temperatures and phase transformations for Edge Sequel Sapphire (a) and Vortex Blue (b)

R-phase to the apparent monoclinic phase starts at -33 °C and finishes at -47 °C. Upon heating, all the martensite converted to austenite starting around 26 °C and finishing around 30–35 °C.

VB also showed classic R-phase transformations. Upon cooling, the austenite to R-phase transformation occurred roughly at 30 °C and an R-phase to martensite transformation [presumable monoclinic] took place upon further cooling around -60 °C. Upon heating, the martensite to R-phase transformation occurred around 20 °C preceding the R-phase to austenite transformation, which took place around 30 °C.

The findings for mean life behavior at the two different tested temperatures correlated with the different martensitic/ austenitic transformation temperatures found in the DSC analysis. Both instruments, VB and SP, would likely be in an austenitic state at body temperature both if coming to 37 °C from above or from below. At the same time, SP would be in a martensitic state at ambient temperature if coming from below, but in a mixed transformational state if coming from above. On the contrary, VB would be in an R-phase state at ambient temperature both if coming from above and from below.

Discussion

Manufacturers in the field of endodontics continue to introduce new strategies to enhance the properties of rotary instruments. CF resistance of NiTi rotary instruments has been of concern since they were first introduced [7], although there are currently still no standardized specifications to address this topic.

Research related to CF resistance of endodontic instruments has improved in recent years. It has been shown that intracanal temperature ranges from 31 to 35 °C [16] within 30 to 60 s after deposition [17]. Studies have also shown that CF of endodontic instruments is significantly reduced at body temperature compared to testing in air at room temperature [8–11], although in static models that disregard the changes in temperature that a rotary instrument may experience when moving in and out the root canal. Accordingly, our results showed a marked decrease in the fatigue life of all tested instruments when tested at body temperature.

A recent study [10] compared files at four different temperatures: 3, 22, 37, and 60 °C. The reason for choosing different temperatures in that study was based on the possibility of clinical use of heated or cooled irrigant solutions. The use of heated irrigant solution has been previously suggested to respectively improve the disinfection and tissue dissolution [19]. However, it should be considered that clinically, irrigant temperature may quickly equilibrate to body temperature after placement in the root canal [16]. Moreover, it has been reported that the benefit of heated irrigants only lasts while they are being delivered, since body temperature is reached immediately after. In the same way, an irrigant used at room temperature is quickly warmed to body temperature when entering the root canal [18].

Others offered the use of cooled irrigant solution as a mean to reduce inflammation in the periradicular region [20]; however, the cooled solution was delivered after the shaping procedure was finalized [21]. Therefore, NiTi rotary instrumentation would still commence at body temperature when using cold irrigation solutions to reduce inflammation [22]. For these reasons, the present study selected only room (21 °C) and body (37 °C) temperatures.

Heat treatment applied to NiTi alloy, to the wire blank either before instrument manufacture or after grinding has an effect on CF resistance [22–24]. For example, ProTaper Gold instruments [Dentsply Sirona] were found to have increased fatigue resistance and flexibility compared to ProTaper Universal instruments, manufactured with the exact same design but made of traditional NiTi alloy, when tested at room [9, 23] and body temperature [11]. Moreover, the resistance of ProTaper Gold files was not significantly affected when tested at body temperature, which has been attributed to an austenite finish temperature higher than 37 °C [11].

In the present study, all files showed a reduced fatigue life when tested at body temperature. This is in accordance with a previous report for Vortex Blue size no. 25.06 [11]. However, in the present study, the reduction of fatigue life for VB instruments at body temperature was 50% for the smaller diameters nos. 20, 2,5 and 30] but less for the larger diameters [no. 35 and no. 40]. In contrast, SP files demonstrated a drastic reduction in fatigue life for all tested diameters at body temperature.

There is comparatively sparse information in the literature and from the manufacturer regarding EdgeEndo files. A previous study showed that no. 25.04 taper EdgeFile instruments (although with no reference to the exact Edge instrument) were significantly more resistant to no. 25.04 taper Vortex Blue instruments at different temperatures [10]. The manufacturer claims that SPs are heat-treated files and compatible to some of the available instruments in the market, including VB, and also states that they are two times more fatigue resistant [12]. Apparently, the recommendations for equivalency among the different available systems are mainly related to the dimensions and sequence for clinical use. Nevertheless, the present study tested instruments of several sizes and the results showed that their performance was not "twice as good" compared to VB and indeed different for each file of the tested EdgeEndo files. The increase in temperature affected the fatigue performance of all instruments, to a different extent. A Weibull analysis was performed to show the reliability of each instrument and the probability of fracture at each temperature [24]. For instrument size no. 25 at room temperature, there was no difference between VB and SP.

However, for the other combinations of sizes and temperatures, significant differences were found.

At body temperature, the VB sizes no. 25, no. 30, and no. 40 lasted significantly longer by at least > 70%, compared to SP. On the contrary, SP sizes no. 30, no. 35, and no. 40 exhibited longer fatigue life compared to the corresponding VB instruments when tested at room temperature. Moreover, for the smallest size tested in this study (no. 20), VB instruments significantly outlasted the SP both at room and body temperature. At the same time, size no. 25 files lasted longer than did size no. 20 in both file brands and at both temperatures, although previous studies have shown that the larger the diameter, the lower the fatigue resistance of the endodontic instrument [24–26].

Weibull analysis provided further information, including the predictability of instrument performance. Figure 3 shows the so-called Weibull cumulative distribution function, where the beta parameter (Table 1) is the slope of the line in the Weibull probability plots. The steeper the slope (β), the smaller the variation in the time to failure and the more predictable the instrument [27]; notably, VB showed the highest beta parameter. As an example, the beta parameter for VB size no. 20 at body temperature was 68.2% higher than for SP size no. 20. In the same way, the line for VB at room temperature in the no. 20 plot (Fig. 3) is almost completely vertical, implying better quality control [27].

In addition, data from the DSC analysis further explained the results obtained in the CF test. Two DSC regular exothermic peaks were observed for SP on the cooling cycle indicating a two-stage phase transformation (Fig. 4); however, the instrument showed a drastic absorption of heat for phase transformation during the heating cycle that may correlate with the lower predictability shown in the Weibull analysis.

Conclusions

Taken together, the results from both the CF resistance tests and DSC analysis suggest the following conclusions: Immersion in water at body temperature was associated with a marked decrease in the fatigue life for all rotary instruments tested. Overall, Vortex Blue instruments were significantly more fatigue resistant and showed the highest predictability at body temperature; however, while smaller diameters of Vortex Blue were also significantly more resistant at room temperature, larger diameters (no. 30, no. 35, and no. 40) were less fatigue resistant than Edge Sapphire under those conditions.

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Compliance with ethical standards

Conflict of interest Dr. Ove Peters serves as a consultant for Dentsply Sirona. The remaining authors declare that they have no conflict of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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