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Correlation between Temperature-dependent Fatigue Resistance and Differential Scanning Calorimetry Analysis for 2 Contemporary Rotary Instruments

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Abstract

Introduction: The aim of this study was to assess differences in cyclic fatigue (CF) life of contemporary heat-treated nickel-titanium rotary instruments at room and body temperatures and to document corresponding phase transformations. **Methods:** Forty Hyflex EDM (H-EDM) files (Coltene, Cuyahoga Falls, OH [#25/.08, manufactured by electrical discharge machining]) and 40 TRUShape (TS) files (Dentsply Tulsa Dental Specialties, Tulsa, OK [#25/.06v, manufactured by grinding and shape setting]) were divided into 2 groups (n = 20) for CF resistance tests in a water bath either at room (22°C ± 0.5°C) or body temperature (37°C ± 0.5°C). Instruments were rotated in a simulated canal (angle = 60°, radius = 3 mm, and center of the curvature 5 mm from the tip) until fracture occurred. The motor was controlled by an electric circuit that was interrupted after instrument fracture. The mean half-life and beta and eta Weibull parameters were determined and compared. Two instruments of each brand were subjected to differential scanning calorimetry (DSC). **Results:** While TS instruments lasted significantly longer at room temperature (mean life = 234.7 seconds; 95% confidence interval [CI], 209–263.6) than at body temperature (mean life = 83.2 seconds; 95% CI, 76–91.1), temperature did not affect H-EDM behavior (room temperature mean life = 725.4 seconds; 95% CI, 658.8–798.8 and body temperature mean life = 717.9 seconds; 95% CI, 636.8–809.3). H-EDM instruments significantly outlasted TS instruments at both temperatures. At body temperature, TS was predominantly austenitic, whereas H-EDM was martensitic or in R-phase. TS was in a mixed austenitic/martensitic phase at 22°C, whereas H-EDM was in the same state as at 37°C. **Conclusions:** H-EDM had a longer fatigue life than TS, which showed a marked decrease in fatigue life at body temperature; neither the life span nor the state of the microstructure in the DSC differed for H-EDM between room or body temperature. *(J Endod 2017;■:1–5)*

Significance

Rotary instruments manufactured by electrical discharge machining showed higher fatigue resistance over those made via grinding and shape setting; however, the manufacturing process seemed to decrease predictability. The endothermic and exothermic reactions for martensitic/austenitic transformations correlate with fatigue resistance findings.

In 1988, Walia et al (1) introduced the use of nickel-titanium (NiTi) alloys for endodontic instruments. Since then, NiTi instruments have steadily provided improved properties, including ease of clinical use because of their flexibility (2), efficiency, and cutting abilities (3). However, these instruments undergo repetitive strain excursions (4) rotating in curved canals and hence are prone to unexpected cyclic fatigue (CF) failure (5–7).

Recently, a new era has started in the development of NiTi rotary instruments with the appearance of proprietary thermal treatments. The new goal of manufacturers is to improve the mechanical properties of endodontic instruments through heat treatment in gas atmospheres or salt baths (8). Improved mechanical properties of files manufactured with these novel alloys have been thoroughly reported (8–11), first with the introduction of M-Wire (Sportswire LLC, Langley, OK) and R-phase (SybronEndo, Orange, CA) and later with so-called controlled memory or CM-Wire (DS Dental, Johnson City, TN). More recently, Blue and Gold alloys (Dentsply Sirona, York, PA) have appeared, named for the blue/gold-colored layer that results from the oxidation that the proprietary heating and cooling processes induce in the surface of the instrument. These new, more martensitic alloys have shown better fatigue behavior (9–11).

The manufacturing method is also known to influence the CF resistance of shaping rotary instruments (12); for example, the grinding process used for most instruments introduces microfractures into the wire, whereas the procedure used in the Twisted File (SybronEndo) reportedly avoids such defects (13, 14).

In contrast, HyFlex EDM (H-EDM; Coltene, Cuyahoga Falls, OH) is manufactured from CM alloy via an electrodischarge machining (EDM) process. This is a noncontact thermal erosion process to machine electrically conductive materials using precisely controlled sparks (electrical discharges) that occur between an electrode and a workpiece in the presence of a dielectric fluid. The spark removes material from both the electrode and the workpiece. Material at the closest points between the electrode and the workpiece, where the spark originates and terminates, is heated to the point of material vaporization. The main difference to other machining methods, like the

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abrasive grinding process, is that the electrode does not make physical contact for material removal; therefore, although the electrode may be considered the cutting tool, EDM involves no actual contact (15).

However, superficial analysis conducted by environmental scanning electron microscopy on new EDM files confirmed the presence of a peculiar irregular surface texture derived from the manufacturing process (16). Moreover, high-magnification micrographs showed a nonuniform structure with pits, pores, and voids causing the peculiar aspect of a "rough spark-machined" surface and the typical "craterlike" surface of EDM materials (16).

Finally, TRUShape (TS; Dentsply Tulsa Dental Specialties, Tulsa, OK) conforming instruments (17–19) required a new manufacturing process to generate their characteristic S shape, which is realized by grinding and subsequent shape setting. Shape setting heat treatment is performed by means of jigs and tools to fix a desired conformation.

CF resistance has traditionally been tested at room temperature; however, newer alloys present transformation temperatures much higher than those of conventional austenitic materials (20) that may in fact transform close to body temperature (8). Recent studies have shown drastically different fatigue behavior of rotary instruments when tested at different temperatures (21–23). Specifically, CF resistance of contemporary rotary instruments decreased when tested approximately at body temperature compared with room temperature (21, 22, 24). However, there are no published data regarding the effect of the new EDM manufacturing process in CF resistance at varying temperatures. Therefore, the aim of this study was to test the effect of temperature on the fatigue limits of contemporary heat-treated NiTi rotary instruments and to document the corresponding martensitic/austenitic transformation temperatures.

Materials and Methods

Instruments

Selected sizes of NiTi rotary instruments H-EDM (size #25/.08) and TS (size #25/.06v) were used in this study. A total of 80 instruments (40 H-EDM and 40 TS files) were divided into 4 groups (n = 20 in each) to be tested for CF in a water bath either at room temperature (22°C ± 0.5°C) or at body temperature (37°C ± 0.5°C). No torque limit was applied, and speed (rpm) was set according to the respective manufacturer guidelines (300 rpm for TS and 400 rpm for H-EDM).

CF Platform

A plastic base with 3 adjustable stainless steel pins was used to simulate the curvature of a root canal with an angle of curvature of 60°, a radius of 3 mm, and the center of the curvature at 5 mm from the instrument tip. The radius and angle of curvature were determined according to Pruett et al (25). Each pin was 6 mm in diameter and 4 cm long and contained a 0.5-mm-wide V-shaped notch where instruments were positioned for the test. A 1:8 gear reduction handpiece connected to an electric motor (Promark Endo, Dentsply Tulsa Dental Specialties) was attached to the same plastic base. The plastic base with pins, the instrument, and 2 connected clips were prepositioned inside a glass container filled with 200 mL deionized water acquired from a MilliQ Integral unit (Millipore, Billerica, MA) 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. Room temperature was measured at 22°C ± 0.5°C, and the first set of tests was performed at this temperature. To achieve body temperatures, the glass container was placed on a hot plate, and the water temperature was stabilized at 37°C ± 0.5°C; the second set of tests was performed under that condition. The temperature was constantly monitored during all tests, and sufficient time after immersion of the base was allowed to equilibrate temperatures.

The motor was controlled by 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 second) at the same time. This design allowed the interruption of the electric circuit linked between the pins, motor, and microcomputer when the instrument fractured during the cyclic fatigue test. This, in turn, stopped the electronic stopwatch, whose time was registered for subsequent Weibull analysis.

Differential Scanning Calorimetry Analysis

Two instruments of each brand were tested using differential scanning calorimetry (DSC) analysis (with scans ranging from approximately 90°C to −60°C) to assess transformation temperatures and phase transformations. Sample preparation consisted on manual diagonal cutting of each instrument in short sections (length = 1–4 mm/weight = 10–20 mg) starting at the tip. Sections were weighed to an accuracy of ±0.01 mg before being placed in a preweighed Tzero aluminum pan (TA Instruments, New Castle, DE). 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 3 times per sample. 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' sigmoidal tangent method.

Weibull Analysis

Weibull analysis (Weibull ++ 7; Reliasoft Corporation, Tucson, AZ) was used to calculate the following parameters (and their 95% confidence intervals [CIs]) for each group:

1. The mean life (seconds) or the expected or average time to failure
2. Beta or 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), also known as the characteristic life or scale parameter (ie, expected time that 63.2% of the files will attain without breakage)

Comparison between the groups determined whether items from one set would outlast those of the others.

Results

CF

Weibull probability plots (reliability vs time) per group are shown in Figure 1. The mean life, eta and beta parameters, and their 95% CIs are shown in Table 1.

TS instruments lasted significantly longer when tested at room temperature (mean life = 234.7 seconds; 95% CI, 209–263.6) compared with body temperature (mean life = 83.2 seconds; 95% CI, 76–91.1). In contrast, temperature did not affect H-EDM behavior (room temperature mean life = 725.4 seconds; 95% CI, 658.8–798.8 and body temperature mean life = 717.9 seconds; 95% CI, 636.8–809.3). H-EDM instruments significantly outlasted TS instruments at both temperatures with a probability of 100%. On the other hand, the highest beta parameter