Effect of autoclave sterilization on the cyclic fatigue resistance of thermally treated Nickel–Titanium instruments

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Abstract

Aim To compare the cyclic fatigue resistance of HyFlex CM, Twisted Files (TF), K3XF, Race, and K3, and evaluate the effect of autoclave sterilization on the cyclic fatigue resistance of these instruments both before and after the files were cycled.

Methodology Five types of NiTi instruments with similar size 30, .06 taper were selected: HyFlex CM, TF, K3XF, Race and K3. Files were tested in a simulated canal with a curvature of 60° and a radius of 3 mm. The number of cycles to failure of each instrument was determined to evaluate cyclic fatigue resistance. Each type of instruments was randomly divided into four experimental groups: group 1 (n = 20), unsterilized instruments; group 2 (n = 20), pre-sterilized instruments subjected to 10 cycles of autoclave sterilization; group 3 (n = 20), instruments sterilized at 25%, 50% and 75% of the mean cycles to failure as determined in group 1, and then cycled to failure; group 4 (n = 20), instruments cycled in the same manner as group 3 but without sterilization. The fracture surfaces of instruments were examined by scanning electron microscopy (SEM).

Results HyFlex CM, TF and K3XF had significantly higher cyclic fatigue resistance than Race and K3 in the unsterilized group 1 (P < 0.05). Autoclave sterilization significantly increased the MCF of HyFlex CM and K3XF (P < 0.05) both before and after the files were cycled. SEM examination revealed a typical pattern of cyclic fatigue fracture in all instruments.

Conclusions HyFlex CM, TF and K3XF instruments composed of new thermal-treated alloy were more resistant to fatigue failure than Race and K3. Autoclaving extended the cyclic fatigue life of HyFlex CM and K3XF.

Keywords: autoclave sterilization, cyclic fatigue, NiTi instrument, thermal treated.

Received 14 November 2014; accepted 9 September 2015

Introduction
The fracture of instruments used in rotary motion occurs in two ways: torsion and flexural cyclic fatigue (Sattapan et al. 2000, Plotino et al. 2009, Shen & Cheung 2013). Cyclic fatigue fracture is caused by repeated tension/compression cycles accumulated at the point of maximum flexure in a curved canal. To reduce the risk of intracanal fracture caused by cyclic fatigue, new file designs and manufacturing methods have been developed. Thermal processing procedures which optimize the microstructure of NiTi alloys are one of the most fundamental approaches that affect the fatigue resistance of NiTi endodontic files (Shen et al. 2012, 2013a,b,c). CM Wire (DS Dental, Johnson...
Thus, this study aimed to compare the cyclic fatigue resistance between thermally treated and traditional NiTi instruments, as well as to evaluate the effect of autoclave sterilization on the cyclic fatigue resistance of these instruments. The null hypothesis is that there is no difference in fatigue resistance between thermally treated and conventional NiTi files, and autoclave sterilization does not affect their fatigue resistance.

Materials and methods

Three NiTi rotary endodontic instruments made from thermal-treated NiTi alloy [HyFlex CM (Coltene Whaledent), TF and K3XF (SybronEndo)] and two NiTi rotary endodontic instruments made from traditional NiTi alloy [Race (FKG Dentaire, La-Chaux-de-Fonds, Switzerland) and K3 (SybronEndo)] with same size 30, .06 taper were selected. The effect of autoclave sterilization on the NCF was tested both before and after the files were cycled. Each type of instruments was randomly divided into four experimental groups. For group 1, files \( n = 20 \) in each group were tested without sterilization to establish baseline mean cycles to failure (MCF). For group 2, files \( n = 20 \) in each group were tested after 10 cycles of autoclave sterilization (Melag Autoklav Vacuklav 23 B+, Melag oHG, Germany). Each cycle was performed including sterilization at 134 °C, with a pressure of 30 psi, for 20 min and then dying for 15 min (Plotino et al. 2012). For group 3, files \( n = 20 \) in each group were autoclaved at 25%, 50% and 75% of the mean cycles to failure (MCF) determined in group 1. All remaining files were then cycled to failure (Hilfer et al. 2011). The autoclave sterilization was performed at 134 °C, with a pressure of 30 psi, for 5 min (Mize et al. 1998). For group 4, files \( n = 20 \) in each group were cycliced in the same manner as group 3 without sterilization.

All the files were subjected to cyclic fatigue tests in a metal block with a 1.5-mm-wide simulated canal with 60° curve and a 3 mm radius (Fig. 1). The block with dimensions of 36.8 mm × 25.4 mm × 9.5 mm was covered with a swivelling acrylic face to observe the files rotating in the canal and to remove broken files during the test. The canal space was filled with synthetic oil (VV Lubricant No. 56; Whip Mix Corporation, Louisville, KY, USA) to reduce friction. All files were placed in the simulated canal to 19 mm.

A 1:16 reduction contra-angle handpiece (ATR Tecnika Vision, Dentsply Tulsa Dental, Tulsa, OK, USA) was mounted on an Instron Testing Machine.
(Mechanical Tester 8841; Instron Corp., Canton, MA, USA), allowing a precise and reproducible placement of each instrument in the canal. The instruments were rotated until failure occurred at the speed and torque recommended by the manufacturers. HyFlex CM and TF were rotated at 500 rpm, whereas the other files were rotated at 300 rpm. Instrument fracture was detected visually. The time to failure was measured to the 1/100 s using a digital stopwatch (Timex, Middlebury, CT, USA). For each instrument, the time to failure was multiplied by the number of rotations per minute to obtain the NCF.

After cyclic fatigue tests were performed, all of the broken instruments were ultrasonically cleaned in acetone for 5 min to remove oil and then treated with absolute alcohol for 5 min to remove water. The fractured surfaces of the instruments were observed under scanning electron microscopy (SEM) (FEI Sirion FEG, Eindhoven, the Netherlands) to evaluate the topographic characteristics of the fractured surfaces.

Statistical analysis
The number of cycles to failure for the various groups was analysed by ANOVA in SPSS 11.0 (SPSS Inc., Chicago, IL, USA). Means and standard deviations of NCF were calculated for each system. Tukey’s post hoc analysis was applied to determine significant differences between groups. Statistical significance was set at a confidence level of 95%, $P < 0.05$ was considered to be statistically significant.

Results
The number of instruments sterilized at 25%, 50% and 75% of the mean cycles to failure (MCF) is shown in Table 1. Some instruments fractured during the process of pre-cycling to 50% and 75% of MCF (Table 1). The data of cycles to failure for the instruments in the four groups are summarized in Table 2. Unsterilized HyFlex CM, TF and K3XF had significantly higher cyclic fatigue resistance than Race and K3 (group 1) ($P < 0.05$) (Table 1). A significant difference was observed between unsterilized (group 1) and pre-sterilized (group 2) files for HyFlex CM and K3XF ($P < 0.05$), whilst other instruments were not significantly different ($P > 0.05$) for mean NCF (Table 2). Furthermore, a significantly higher mean NCF ($P < 0.05$) was observed for HyFlex CM and K3XF files sterilized before use and after the three periods (25%, 50% and 75%) of cycling (group 3) than for the same files sterilized only after the periods of cyclic stress (group 4). SEM images of the fracture surfaces with crack initiation and dimple regions for each type of instrument are shown in Fig. 2.

Discussion
The two scenarios, multiple sterilizations of unused instruments and repeated sterilization of files after being challenged by stepwise increased amount of cyclic stress (fatigue), were examined in the current study. Sterilization of rotary NiTi files must be done before...
use (except for pre-sterilized files), and this is repeated if the files are reused and/or carried forward between appointments of the same patient. Many practitioners prefer having pre-arranged sets of selected files, all of which may not be used during a single treatment. As a result, unused rotary files are also subjected to multiple autoclave cycles. Most of the previous studies focused on evaluating the effect of repeated sterilization on unused instruments (Mize et al. 1998, Viana et al. 2006, Valois et al. 2008, Plotino et al. 2012).

Only one study (Hilfer et al. 2011) observed the effects of multiple sterilization on cyclic fatigue of thermally treated NiTi instruments (TF and GTX), which were exposed to defined controlled stresses.

A metal block with simulated canals (Fig. 1) was used to evaluate fatigue resistance. The canal had two straight portions with a 60° curve with a small radius between them. Whilst the simplified model in the metal groove cannot represent complicated clinical situations, it has been used in studies to standardize the experimental conditions for cyclic fatigue testing and allows comparisons with previous studies.

In the present study, the fatigue resistance of thermally treated, unsterilized NiTi instruments (HyFlex CM, K3XF and TF) was higher than that of superelastic NiTi instruments (K3 and Race). This is in accordance with several recent studies (Larsen et al. 2009, Gambarini et al. 2011, Bouska et al. 2012, Plotino et al. 2012, Shen et al. 2013b). Interestingly, in the present study, the three thermally treated NiTi instruments without sterilization were equally resistant to cyclic fatigue.

Resistance to cyclic fatigue of both HyFlex CM and K3XF instruments that had not undergone cyclic stress increased significantly after sterilization (10 ster. cycles). No corresponding increase was detected with the TF files and the superelastic NiTi instruments in the present study. The literature of the impact of sterilization on conventional superelastic NiTi instruments appears contradictory. Viana et al. (2006) reported that five autoclave cycles significantly increased the cyclic fatigue resistance of ProFile instruments. Later, Zinelis et al. (2007) reported that sterilization in an autoclave below 170 °C did not provide enough energy to enable a change in the crystalline phase. Plotino et al. (2012) reported that repeated cycles (10 cycles) of autoclave sterilization (134 °C for 35 min) did not influence the mechanical properties of K3, Mtwo (VDW, Munich, Germany) and Vortex (Dentsply Tulsa Dental Specialties) NiTi instruments. However, the K3XF prototypes demonstrated a significant increase in

| Table 1 Baseline scores of fatigue lifespan [the mean cycles to failure (MCF)] for all NiTi instruments, as well as the number of instruments sterilized at 25%, 50% and 75% of the MCF |
|----------------|----------------|----------------|----------------|----------------|
|                | HyFlex CM      | K3XF           | K3             | Race           |
|                | MCF            | MCF            | MCF            | MCF            |
|                | No sterilization | 679 ± 135      | 684 ± 104      | 316 ± 124      | 655 ± 532      |
|                | 25% of MCF     | 20 20 20       | 30 30 30       | 20 20 20       | 30 30 30       |
|                | 50% of MCF     | 20 20 20       | 30 30 30       | 20 20 20       | 30 30 30       |
|                | 75% of MCF     | 510            | 713            | 476            | 85b            |
| Group 3: G3    | 135a           | 634            | 655            | 53b            |
| Group 4: G4    | 20             | 18             | 17             | 18             |
| N: the number of instruments. Different superscript letters indicate statistically different between groups (P < 0.05). |
cyclic fatigue resistance after autoclaving in the same study, which was also confirmed by the present study. Although R-phase is found on both K3XF and TF files, only the TF instruments undergo a twisting process during their manufacture. It is thus possible that the subsurface microstructure of the thermally treated NiTi instruments manufactured by grinding (vs. twisting) before the cyclic stress may be altered by the sterilization process.

In the present study, autoclave sterilization after cyclic stress significantly improved the cyclic fatigue behaviour of HyFlex CM and K3XF files, whereas TF, K3 and Race were not affected (size 30, .06 taper). Hilfer et al. (2011) reported a significant decrease of fatigue resistance after autoclaving at 25%, 50% and 75% of the established mean cycles to failure for TF (size 25, .06 taper). However, size 25, .04 taper TF instruments in the same study were not significantly

Table 2 The number of cycles to failure for instruments that were not sterilized at any point of the experiment (group 1) instruments sterilized 10 times before any use (group 2), instruments sterilized at 0%, 25%, 50% and 75% of the MCF (group 3) and instruments sterilized at 25%, 50% and 75% of the mean cycles to failure (group 4) (Mean ± SD)

<table>
<thead>
<tr>
<th></th>
<th>HyFlex CM</th>
<th>Twisted Files</th>
<th>K3XF</th>
<th>K3</th>
<th>Race</th>
</tr>
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<tbody>
<tr>
<td>No sterilization (group 1)</td>
<td>679 ± 135&lt;sup&gt;a,d&lt;/sup&gt;</td>
<td>634 ± 104&lt;sup&gt;a&lt;/sup&gt;</td>
<td>655 ± 124&lt;sup&gt;a&lt;/sup&gt;</td>
<td>396 ± 53&lt;sup&gt;b&lt;/sup&gt;</td>
<td>409 ± 85&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Sterilization 10 times before cyclic stress (group 2)</td>
<td>849 ± 148&lt;sup&gt;c&lt;/sup&gt;</td>
<td>637 ± 117&lt;sup&gt;a&lt;/sup&gt;</td>
<td>784 ± 162&lt;sup&gt;c&lt;/sup&gt;</td>
<td>407 ± 83&lt;sup&gt;b&lt;/sup&gt;</td>
<td>413 ± 98&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Sterilized at start and after each cyclic stress (group 3)</td>
<td>723 ± 38&lt;sup&gt;c&lt;/sup&gt;</td>
<td>615 ± 75&lt;sup&gt;a&lt;/sup&gt;</td>
<td>718 ± 53&lt;sup&gt;c&lt;/sup&gt;</td>
<td>358 ± 42&lt;sup&gt;b&lt;/sup&gt;</td>
<td>391 ± 75&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>No sterilization at start, only after each cyclic stress (group 4)</td>
<td>656 ± 51&lt;sup&gt;a&lt;/sup&gt;</td>
<td>621 ± 68&lt;sup&gt;a&lt;/sup&gt;</td>
<td>634 ± 48&lt;sup&gt;a&lt;/sup&gt;</td>
<td>370 ± 62&lt;sup&gt;b&lt;/sup&gt;</td>
<td>383 ± 70&lt;sup&gt;b&lt;/sup&gt;</td>
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Different superscript letters indicate statistically significant differences between groups (P < 0.05).

Figure 2 Scanning electron micrographs of the fracture surfaces of (a) TF (×200), (b) Race (×150), (c) K3XF (×150), (d) K3 (×150), (e) HyFlex CM (×150) and instruments with the region of fatigue crack propagation and dimple area outlined (dotted line) with crack origins (arrows).
affected by the same conditions (Hilfer et al. 2011). The different results between the present study and that of Hilfer et al. (2011) may be explained by different instrument size, sterilization condition (132 °C for 6 min vs. 134 °C for 5 min) and fatigue test set-up (90° and a 5 mm curvature radius vs. 60° and a 3 mm curvature radius). The instruments were subjected to repeated, reverse torsional loads and cyclic fatigue stresses inside the canal at the same time. The data obtained here cannot be directly extrapolated to clinical conditions, and any conclusions from the present study must be drawn with caution.

Conclusions

Autoclave sterilization before use of HyFlex CM, TF and K3XF, significantly increased their resistance to cyclic fatigue, whilst no effect was detected with Race and K3. The autoclave sterilization of HyFlex CM and K3XF files after being challenged by cyclic fatigue significantly extended their remaining cyclic fatigue lifetime.

Acknowledgement

This study was financially supported by grants from Hubei Natural Science Foundation (no. 2014CFB468) and the Fundamental Research Funds for the Central Universities (no. 2042015k0103) of China.

References


