FATIGUE BEHAVIOR OF NICKEL-TITANIUM SUPERELASTIC WIRES AND ENDODONTIC INSTRUMENTS

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ABSTRACT
Modern endodontic files are made of Ni-Ti superelastic alloys and can be employed in rotary techniques for the chemical-mechanical preparation of curved root canals. These instruments are usually operated at rotation speeds between 250 and 350rpm, under geometrical conditions causing tensile-compressive cyclic strains with maximum amplitudes in the range of 3 to 5%. To contribute to the knowledge of the fatigue behavior of this material under such high deformation conditions is the aim of this work. Cyclic load-unload tensile tests were performed on NiTi superelastic wires employed in the manufacture of ProFile endodontic instruments. Fatigue tests of new ProFile instruments simulating the geometrical conditions found in their clinical use to format curved root canals were employed to evaluate their fatigue behavior. The NiTi wires, taken from the production line of ProFile instruments just before the final machining step, were tensile-tested to rupture in the as-received condition and after 100 load-unload tensile cycles in the superelastic regime (4% elongation). It was found that only small changes took place in the parameters describing the mechanical behavior of the wires after 100 load-unload cycles. The stress at maximum load and the plastic strain at breakage remained practically the same, while the critical stress to induce martensite decreased by about 26.5%. Analysis of the fracture surface of the cycled wires indicated that the failure of these specimens in tension involved pre-existing fatigue cracks, developed during load-unload cycling. The fatigue tests of ProFile instruments demonstrated that the maximum tensile strain amplitude at the instrument’s surface determines the average number of cycles to failure. The characteristics of the fracture surfaces of the instruments and wire specimens indicate that the nucleation of secondary cracks is the dissipation mechanism responsible for the relatively high fatigue resistance of superelastic NiTi alloys.

1 INTRODUCTION
The use of NiTi superelastic wires to manufacture machine-driven endodontic files constitutes an important development of the endodontic therapy, leading to the application of rotary techniques for the chemical-mechanical preparation of curved root canals. However, when the endodontic instrument rotates inside a curved root canal, it is submitted to tensile-compressive strain cycles, which can give rise to fracture by fatigue (Pruett [1], Melo [2]). Strain levels attained during this cyclic loading depend on the root canal and instrument geometry, being concentrated at the portion of the instrument positioned in the maximum curvature region of the root canal. Among the two parameters generally employed to define the root canal geometry, radius and angle of curvature, the former is the most meaningful, insofar as fatigue resistance of machine driven NiTi instruments is concerned, since the tensile strain component is inversely proportional to this parameter (Pruett [1]). Furthermore, the importance of the geometrical factor in root canal shaping becomes even greater when multiple curvatures are present. The high incidence of secondary curvatures in human lower molars (30%) and the fact that they occur predominantly in the apical third of the root canal, at a mean distance of 2.2mm from the foramen (Cunningham [3]), demand that NiTi rotary endodontic instruments possess exceptional fatigue resistance at relatively high strain levels.

Detailed knowledge of how such instruments behave under fatigue is fundamentally important to ensure that their clinical usage be safe. Therefore, the aim of this study was to obtain basic
information on the mechanical behavior under cyclic loading of the NiTi wires employed in the manufacture of ProFile endodontic instruments. These machine-driven files, produced in Switzerland, are widely used in the endodontic therapy in many countries.

**EXPERIMENTAL PROCEDURE**

The NiTi wires used in this work were provided by Dentsply-Maillefer (Baillagues, Switzerland) and were taken from the production line of ProFile rotary endodontic instruments just before the final machining step. The ProFile instruments used to evaluate the fatigue resistance were obtained from the regular suppliers, withdrawn from sealed boxes and sequentially numbered on the handle, using a high-speed diamond bur. The analyzed sample consisted of ten sets of new files identified by their size and taper. Three sizes were employed: 20, 25 and 30. These numbers correspond to the diameter of the instrument’s tip, in tenth of millimeters. Taper, or conicity, is a parameter indicating how the diameter of the instrument changes from the tip upwards its active length. Two tapers were employed: 0.04 and 0.06, meaning that the instruments diameter increased by 4% or 6% in each millimeter of their length. One set of instruments means ten instruments of the same size and taper. A total of sixty instruments were thus employed, combining size and taper as follows: 20/.04, 25/.04, 30/.04, 20/.06, 25/.06 and 30/.06. These are the files which may fail by fatigue during curved root canals formatting by the rotary technique.

Specimens of the NiTi wires in the as-received condition, with 1.2mm in diameter and 80mm in length, were tensile tested in a universal testing machine (Instron 5581, Canton, MA, USA). The transformation stress, the stress at maximum load (ultimate tensile strength) and the plastic strain at breakage (total elongation) were determined as the average values of three tests performed at room temperature and at a strain rate of $1.0 \times 10^{-3}\text{s}^{-1}$, using an extensometer. Cyclic loading tests were also carried out at room temperature, in similar NiTi specimens and at the same testing machine, at a strain rate of $1.0 \times 10^{-2}\text{s}^{-1}$. The specimens were loaded in the superelastic regime to 4% total elongation and unloaded to zero stress. This cycle was performed 100 times and then the specimens were tensile-tested to failure at a strain rate of $1.0 \times 10^{-3}\text{s}^{-1}$. As before, transformation stress, stress at maximum load and plastic strain at breakage of the cycled wires were evaluated as the average of three tests.

The fatigue resistance of the ProFile instruments was tested in a laboratory fatigue test bench, using an endodontic motor operating at 250rpm and an artificial canal with 5mm radius of curvature, made with quenched H13 tool steel, to avoid geometric changes due to wear. The parameter used to evaluate the fatigue resistance was the average number of cycles to failure – NCF, calculated multiplying the time to failure by the rotation speed. Fracture surfaces were analyzed by scanning electron microscopy – SEM (Jeol JSM 6360, Tokyo, Japan).

**RESULTS AND DISCUSSION**

The average stress-strain curves obtained from three specimens of the NiTi wires in the as received condition and from three specimens previously submitted to 100 load-unload cycles are shown in Figure 1. The stress peak at the beginning of the superelastic plateau corresponds to the nucleation of martensite variants in austenite, while the subsequent decrease in stress is associated with the propagation of these convenient orientated martensite variants (Huang [4]). The mean values of the transformation stress, $\sigma_{AM}$, stress at maximum load (ultimate tensile strength, $\sigma_{UTS}$) and plastic strain at breakage (total elongation, $e_T$), determined for the as received and cycled wires, are shown in Table 1. Comparison of the values of these parameters indicates that only small changes in the mechanical behavior of the wires took place after 100 load-unload cycles up to 4% tensile strain. The stress at maximum load and the plastic strain at breakage are practically the same (the change in $e_T$ is of the order of the standard deviation). In fact, the main effect of cyclic loading was
decreasing $\sigma_{A-M}$ by about 26.5% and increasing the strain at the superelastic plateau by approximately 25%. Tolomeo [5], comparing the mechanical properties of superelastic NiTi stents under monotonic and cyclic loading, found that stress changes due to cyclic loading took place mainly at the superelastic plateau, in agreement with the results found in the present work.

Table 1: Mean values of the transformation stress, $\sigma_{A-M}$, stress at maximum load (ultimate tensile strength, $\sigma_{UTS}$) and plastic strain at breakage (total elongation, $\varepsilon_T$).

<table>
<thead>
<tr>
<th>Property</th>
<th>Condition</th>
<th>As received</th>
<th>Cycled 100 times</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma_{A-M}$ (standard deviation), MPa</td>
<td>550 (7.5)</td>
<td>404 (21.3)</td>
<td></td>
</tr>
<tr>
<td>$\sigma_{UTS}$ (standard deviation), MPa</td>
<td>1,404 (7.0)</td>
<td>1,403 (18.1)</td>
<td></td>
</tr>
<tr>
<td>$\varepsilon_T$ (standard deviation), %</td>
<td>11.2 (0.9)</td>
<td>12.4 (0.3)</td>
<td></td>
</tr>
</tbody>
</table>

The fracture surfaces of as received and cycled wires tensile-tested to failure display the characteristic cup-and-cone fracture of ductile metals, with a peripheral shear area surrounding the fibrous central region, as exemplified in Figure 2a. The fibrous central region of the fracture surfaces contained dimples and slip lines, as expected for this type of failure. The shear area on the fracture surface of the cycled specimen is shown in higher magnifications in Figure 2b, where the presence of fatigue striations and numerous secondary cracks can be observed. Similar features could not be detected on the shear area of wire specimens tensile-tested to failure in the as-received condition. The presence of fatigue striations in the shear area of the fracture surfaces of the cycled wires indicates that the failure of these specimens in the tensile tests involved pre-existing fatigue cracks, developed during load-unload cycling.

The results of the fatigue tests of the endodontic files are summarized in Figure 3, where it can be observed that the number of cycles to failure decreases as the size and taper of the instrument increase. Similar results were obtained by other authors in studies of the fatigue behavior of ProFile instruments (Haikel [6], Yared [7] and Gambarini [8]).

Figure 1: Average stress-strain curves of the NiTi wires in the as received condition and after 100 load-unload cycles.
Figure 2: Secondary electron images by SEM of the fracture surface of a wire specimen cycled 100 times and then tensile tested to failure.

In the test device employed in this work, the instruments were set to rotate at a fixed position, in such a way that the maximum curvature was always located at 3mm from the instrument’s tip. The knowledge of the instrument’s geometry allowed then the estimation of the tensile strain component at the surface of the instrument placed in region of maximum curvature, $\varepsilon_T$, which is given by:

$$\varepsilon_T = \left( \frac{2R}{D} - 1 \right)^{-1} \quad (1)$$

where $R$ is the curvature radius of the artificial canal and $D$ is the instrument’s diameter in the region of maximum curvature, which, in turn, is the sum of the diameter of its tip, $D_0$, with the distance from the tip, $L$, times the instrument’s taper, $T$, that is, $D = D_0 + LT$. A plot of NCF against the estimated values of $\varepsilon_T$ is shown in Figure 4.

Figure 3: Average number of cycles to failure, NCF, of the ProFile instruments tested in fatigue.
The influence of the tensile strain component on the fatigue behavior of Profile instruments are in agreement with what is generally expected when high strain amplitudes are employed. In other materials, such amplitudes would be far beyond the elastic regime. Thus, the superelastic NiTi alloys exhibit exceptional fatigue resistance at high strains, although, as observed by other authors, they may fail rapidly when the cyclic loading is controlled by stress (Duerig [9]).

The fracture surfaces of the ProFile instruments tested in fatigue showed the typical characteristics of fatigue failure at high stress levels, that is, a small peripheral shear area surrounding a large fibrous central region, as illustrated in Figure 5a. A higher magnification image of the shear area pointed out in Fig. 5a is presented in Figure 5b, showing the presence of fatigue striations and secondary cracks (arrowed).

Figure 4: Change in the NCF with the estimated tensile strain component.

Figure 5: Secondary electron images by SEM of the fracture surface of a 30/.06 ProFile instrument fatigue tested to failure. Arrow in (a) marks the area enlarged in (b). Arrows in (b) indicate secondary cracks.
The common aspect revealed by SEM fractography was the presence of a high density of secondary fatigue cracks at the fracture surfaces of cycled wires tensile tested to failure and of ProFile instruments tested in fatigue. It is possible that the fast and multiple nucleation of secondary cracks, which can be associated with the large amount of martensite variant interfaces and twins generated during the deformation cycle in the superelastic regime (Hornbogen [10]), is the dissipation mechanism responsible for the relatively high fatigue resistance of the superelastic NiTi alloys employed in endodontics.

CONCLUSIONS
The results obtained in this work indicate that NiTi superelastic wires are the most appropriate material to manufacture NiTi endodontic instruments to clean and format curved root canals using the rotary technique. Cyclic load-unload tensile deformation in the superelastic regime has little effect on the material’s mechanical properties. The strain controlled fatigue tests of ProFile instruments analyzed in geometrical conditions similar to those found in the clinical practice demonstrated that these instruments exhibit an exceptional fatigue resistance for the high level of tensile strains they are submitted to. This behavior was associated to the dissipation of mechanical energy associated with nucleation and propagation of secondary fatigue cracks along martensite variant and twin borders formed during cyclic loading superelastic NiTi.

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REFERENCES