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Fatigue behaviour of nickel–titanium superelastic wires and endodontic instruments

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ABSTRACT Endodontic files made of nickel–titanium (NiTi) superelastic wires can be employed in rotary techniques for cleaning and shaping curved root canals, suffering tensile–compressive strain cycles with maximum amplitudes between 3 and 5%. The aim of this work was to study the fatigue behaviour of this material under such high deformation conditions, using NiTi instruments and superelastic wires taken from their production line. One hundred load–unload tensile cycles in the superelastic regime (4% elongation) were applied to NiTi wires. New endodontic instruments were fatigue-tested simulating the geometrical conditions found in their clinical use. It was found that only small changes took place in the parameters describing the mechanical behaviour of the cycled wires. The measured average number of cycles to failure varies inversely with the maximum tensile strain amplitude in the fatigue tests ($r = 0.993$).

Keywords endodontic instruments; fatigue; mechanical behaviour; NiTi wires.

NOMENCLATURE

D = file diameter (mm)
D_0 = file tip diameter (mm)
D_3 = file diameter at 3 mm from the tip (mm)
L = distance from the file tip (mm)
NCF = number of cycles to failure
R = curvature radius of the artificial canal (mm)
T = file taper
ε_P = plastic strain at breakage (%)
ε_T = tensile strain amplitude (%)
$\sigma_{A\text{–}M}$ = transformation stress (MPa)
σ_{UTS} = ultimate tensile strength (MPa)

INTRODUCTION

The use of nickel–titanium (NiTi) superelastic wires to manufacture rotary 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. Regarding modern concepts, the final root canal shape has to allow adequate irrigation and close adaptation of the filling material during obturation. High lateral forces can develop in the preparation of curved canals due to the stiffness of the endodontic instruments, influencing the amount of dentin removed from the canal walls. The resulting canal aberrations include ledges, zippings and perforations. NiTi rotary endodontic instruments were developed to overcome these inconveniences, because they are able to maintain the original canal shape without creating severe irregularities, particularly in narrow curved canals.[1]

The main characteristic of the NiTi alloys used in the manufacture of rotary endodontic instruments is a large nonlinear elasticity that allows a recovery of up to 8% of the tensile strains upon unloading. This is called the superelastic effect and stems from the stress-induced formation of martensite taking place during loading, which, under appropriate conditions, accommodates the applied

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stress. In a stress–strain curve, formation of martensite starts at a given stress level, called the transformation stress, at which the curve departures from linearity. In this aspect, it is similar to the yield point of ordinary metallic materials, but the microscopic changes responsible for this deformation are reversible. When the stress is released, the martensitic phase transforms back into austenite and the material recovers its original dimensions and shape.

Superelastic NiTi alloys are two to three times more flexible than stainless steel, the material employed to manufacture endodontic hand files. This is due to its low elastic modulus, but is also a consequence of the superelastic behaviour of NiTi, because the transformation stress remains approximately constant when the material is transforming to martensite. The restoration stress acting during unloading, when martensite transforms back to austenite, is also approximately constant and smaller than the transformation stress, giving rise to mechanical hysteresis during a load–unload cycle.

In straight root canals, rotary endodontic files operate by cutting and removing organic tissue and debris, experiencing mostly frictional forces opposing their torsional motion. However, when the instrument rotates inside a curved root canal it is bent and thus submitted to tensile–compressive strain cycles in the region of the canal curvature, in addition to the torsional restraints. This alternating strain cycling can give rise to failure of the instruments by fatigue.[2,3] The strain levels attained by the endodontic files during this cyclic loading depend on the root canal and instrument geometries, being concentrated at the portion of the file 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, because the tensile strain component is inversely proportional to this parameter.[2] 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 2.2 mm from the foramen,[4] demand that NiTi rotary endodontic instruments possess exceptional fatigue resistance at relatively high strain levels. When NiTi endodontic instruments are clinically employed for preparing curved root canals using the rotary technique, cyclic bending strains with maximum tensile amplitudes in excess of 5% are developed.[5]

Detailed knowledge of how such instruments behave under fatigue is thus fundamentally important to ensure that their clinical usage be safe. Therefore, the aim of this study was to obtain basic information on the mechanical behaviour under cyclic loading of the NiTi wires employed in the manufacture of endodontic instruments.

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 cutting shaft of these instruments have a conical shape, starting at a non-cutting tip of small diameter and going up at increasing diameters along 16 mm, where the shaft becomes cylindrical. Circular grooves are made in this cylindrical shaft to indicate the size and taper (conicity) of the instrument. It is attached to the instrument's handle, by means of which the file is set to rotate. An instrument size 25, 0.06 taper, usually denoted as 25/.06, is shown in Fig. 1, which is a low-magnification scanning electron microscopy (SEM) image composition (built by superposing two complementary images). Six sets of instruments, each containing 10 instruments of the same size and taper, in a total of 60 instruments, were analysed. Three sizes were employed: 20, 25 and 30. The instrument size corresponds to the diameter of its tip in 10th of millimetres. The tapers analysed were 0.04 and 0.06, meaning that the diameter of the instruments cutting edge increased by 0.04 or 0.06 mm in each millimetre of their length.

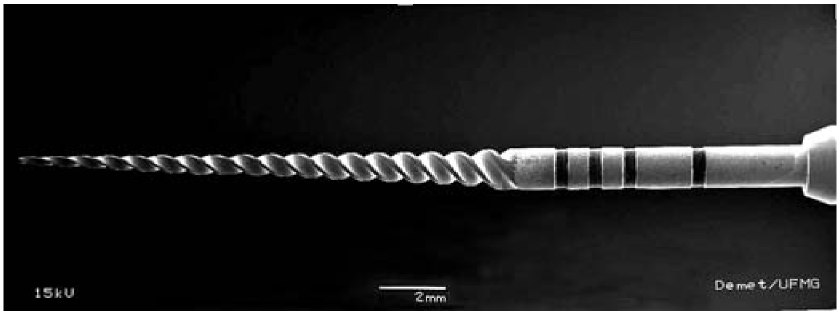

Fig. 1 SEM image composition of an instrument size 25, 0.06 taper.

Specimens of the NiTi wires in the as-received condition, with 1.2 mm in diameter and 80 mm in length, were tensile-tested in an Instron universal testing machine. The transformation stress, $\sigma_{A\text{–}M}$, the stress at maximum load (ultimate tensile strength), σ_{UTS} and the plastic strain at breakage (total elongation), ε_P, were determined as the average values of three tests performed at room temperature and at a strain rate of 1.0×10^{-3} 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 1.0×10^{-2} s^{-1}. The specimens were loaded in the superelastic regime to 4% total elongation and unloaded to zero stress. This is a strain-controlled fatigue test, but unloading to zero strain would lead to plastic deformation of the specimens, because the superelastic strain is not fully recovered after such a high straining level. This cycle was performed 100 times and then the specimens were tensile-tested to failure at a strain rate of 1.0×10^{-3} 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.

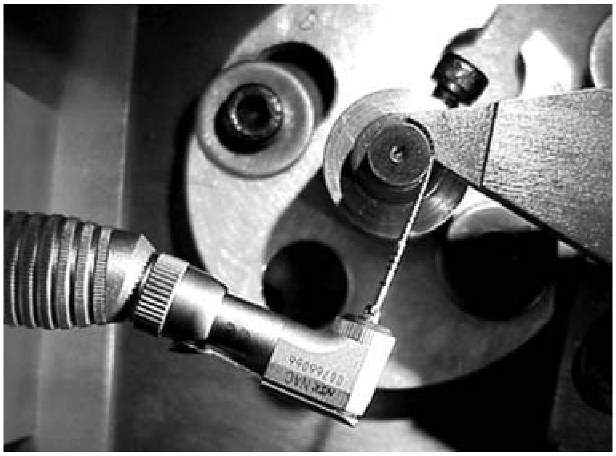

Fig. 2 Artificial canal with an instrument mounted.

The fatigue resistance of the endodontic instruments was tested in a laboratory fatigue test bench, using a endodontic motor operating at 250 rpm and an artificial canal made with quenched H13 tool steel to avoid geometric changes due to wear. Figure 2 shows a file inserted in the artificial canal. Its radius and angle of curvature, 5 mm and 45°, respectively, were chosen based on measurements of these parameters previously performed on curved mesial canals in mandibular human molars and curved buccal canals in maxillary molars.[5] The parameter used to evaluate the fatigue resistance was the average number of cycles to failure (NCF), calculated by multiplying the time to failure and the rotation speed. Fracture surfaces were analysed by SEM (Jeol JSM 6360).

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 Fig. 3. 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.[6] The mean values of $\sigma_{A\text{–}M}$, σ_{UTS} and ε_P, 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

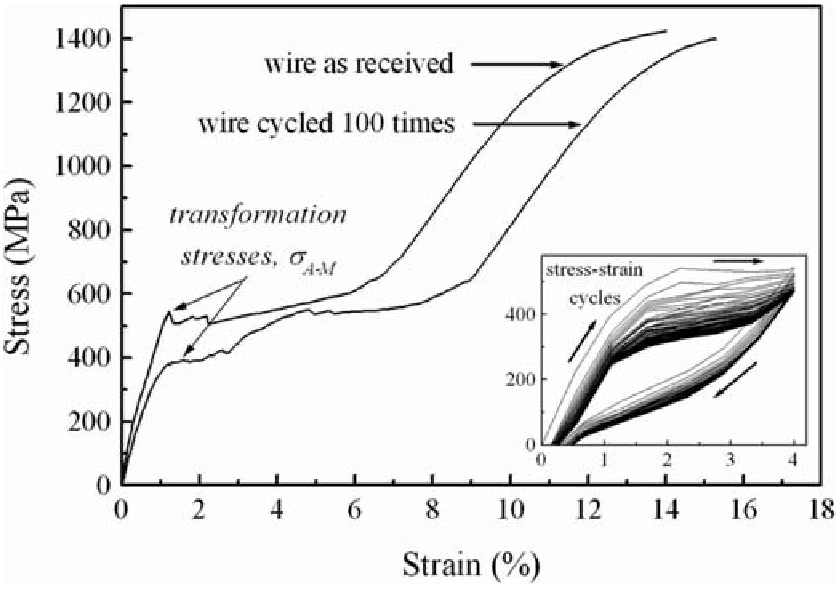

Fig. 3 Average stress–strain curves of the NiTi wires in the as-received condition and after 100 load–unload cycles. The inset shows cycling curves.

Table 1 Mean values of the transformation stress, $\sigma_{\mathrm{A-M}}$, ultimate tensile strength, σ_{UTS} and plastic strain at breakage, ε_{P}

	Condition	
Property	As-received	Cycled 100 times
$\sigma_{\mathrm{A-M}}$ (standard deviation), MPa	550 (7.5)	404 (21.3)
σ_{UTS} (standard deviation), MPa	1404 (7.0)	1403 (18.1)
ε_{P} (standard deviation), %	11.2 (0.9)	12.4 (0.3)

behaviour of the wires took place after 100 load–unload cycles up to 4% tensile strain. The ultimate tensile strength and the plastic strain at breakage are practically the same (the change in ε_{P} is of the order of the standard deviation). In fact, the main effect of cyclic loading was decreasing $\sigma_{\mathrm{A-M}}$ by about 26.5% and increasing the strain at the superelastic plateau by approximately 25%. The cycling behaviour illustrated by the inset in Fig. 3 corroborates these observations, showing also that the unrecoverable strain increases, while the hysteresis loop decreases, as the number of cycles is increased. Tolomeu *et al.*,[7] 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.

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. The transition area of the fracture surface is illustrated in Fig. 4. The fibrous central region of the fracture surfaces contained dimples and slip lines, as expected for this type of failure. The presence of fatigue striations and numerous secondary cracks on the shear area of a previously cycled specimen can be observed in Fig. 4b. Similar features could not be detected on the shear area of wire specimens tensile-tested to failure in the as-received condition (Fig. 4a). The fatigue striations in the shear area of the fracture surfaces of the cycled wires indicate that the failure of these specimens in the tensile tests involved pre-existing fatigue cracks, developed during load–unload cycling.

In the fatigue 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 3 mm 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, ε_{T}, which is given by

$$\varepsilon_{\mathrm{T}} = \left(\frac{2R}{D} - 1\right)^{-1} \tag{1}$$

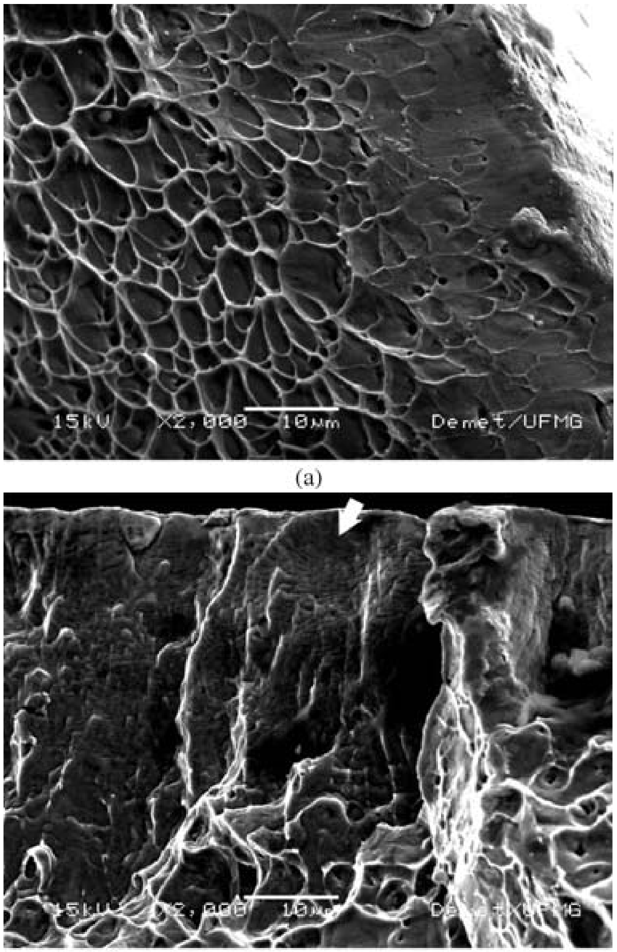

Fig. 4 SEM images of the fractured surface of (a) an as-received and (b) a cycled wire tensile-tested to failure. Arrow in (b) indicates fatigue striations and secondary cracks.

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, $D = D_0 + LT$. The results of the fatigue tests of the endodontic files are summarized in Fig. 5 in terms of the NCF as a function of the instrument diameters at 3 mm from the tip, D_3. It can be observed that the NCF decreases as D_3, and thus the size and taper of the instruments, increase. Similar results were obtained by other authors in the studies of the fatigue behaviour of ProFile instruments.[8–10]

A plot of NCF against the estimated values of ε_{T} is shown in Fig. 6. The measured average NCF varies inversely with the maximum tensile strain amplitude in the fatigue tests ($r = 0.993$). The influence of the tensile strain component on the fatigue behaviour is in agreement with what is generally expected when high strain amplitudes are

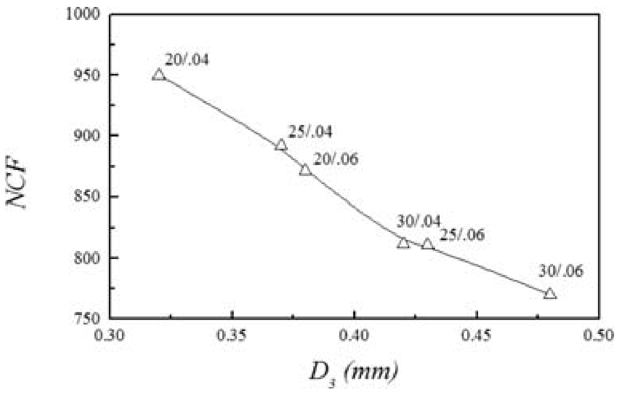

Fig. 5 Average, NCF, as a function of instrument diameter at 3 mm from the tip, D_3.

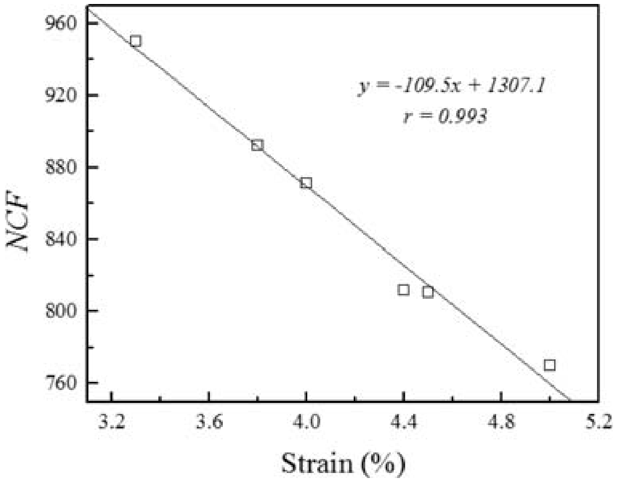

Fig. 6 Change in the NCF with the estimated tensile strain component.

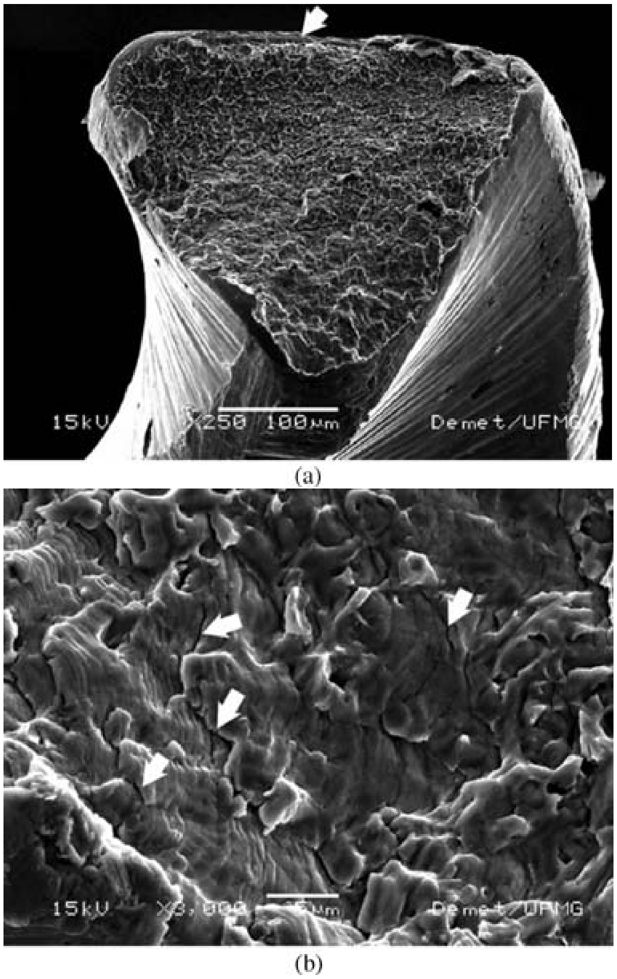

Fig. 7 SEM images of the fractured surface of a 30/.06 ProFile instrument fatigue-tested to failure. Arrow in (a) marks the area enlarged in (b). Arrows in (b) indicate secondary cracks.

employed. In other materials, such amplitudes would be far beyond the elastic regime. Although the expected fatigue lives of the tested instruments seem small ($<10^3$ cycles), it is important to mention that they are usually employed to format only 10 curved root canals before being discarded. In a previous work, it was shown that the fatigue life consumption of these instruments during shaping 10 curved root canals corresponds to an average of 58% of their expected fatigue life.[5]

The fracture surfaces of the 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 Fig. 7a. A higher magnification image of the shear area pointed out in Fig. 7a is presented in Fig. 7b, showing the presence of fatigue striations and secondary cracks (arrowed). 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 the endodontic 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,[11] is the dissipation mechanism responsible for the relatively high fatigue resistance of the superelastic NiTi alloys employed in endodontics.

CONCLUSIONS

Cyclic load–unload tensile deformation in the superelastic regime has little effect on the material's mechanical properties. The strain-controlled fatigue tests of the analysed endodontic instruments demonstrated that they exhibit an exceptional fatigue resistance for the high level of tensile strains usually found in practice. This behaviour was associated to the dissipation of mechanical energy due

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