

Fatigue Testing of Controlled Memory Wire Nickel-Titanium Rotary Instruments

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Abstract

Introduction: To improve the fracture resistance of nickel-titanium (NiTi) files, manufacturers have introduced new alloys to manufacture NiTi files and developed new manufacturing processes. This study was aimed to examine the fatigue behavior of NiTi instruments from a novel controlled memory NiTi wire (CM Wire). **Methods:** Instruments of ProFile, Typhoon (TYP), Typhoon CM (TYP CM), DS-SS0250425NEY (NEY), and DS-SS0250425NEY CM (NEY CM) (DS Dental, Johnson City, TN) all size 25/.04 were subjected to rotational bending at the curvature of 35° and 45° in air at the temperature of 23° ± 2°C, and the number of revolutions to fracture (N_f) was recorded. The fracture surface of all fragments was examined by a scanning electron microscope. The crack-initiation sites, the percentage of dimple area to the whole fracture cross-section, and the surface strain amplitude (ϵ_s) were noted. **Results:** The new alloy yielded an improvement of over three to eight times in N_f of CM files than that of conventional NiTi files ($P < .05$). The vast majority of CM instruments (50%-92%) showed multiple crack origins, whereas most instruments made from conventional NiTi wire (58%-100%) had one crack origin. The values of the fraction area occupied by the dimple region were significantly smaller on CM NiTi instruments compared with conventional NiTi instruments ($P < .01$). The square (NEY CM) versus the triangular (TYP CM) configuration showed a significantly different lifetime on CM wire at both curvatures ($P < .01$). **Conclusions:** The material property had a substantial impact on fatigue lifetime. Instruments made from CM Wire had a significantly higher N_f and lower surface strain amplitude than the conventional NiTi wire files with identical design. (*J Endod* 2011;37:997-1001)

Key Words

Controlled memory, fatigue, nickel-titanium instrument, ProFile

The development of nitinol, an equiatomic alloy composed of nickel and titanium (1), has proved to be a significant advancement in the manufacture of endodontic instruments. Nickel-titanium (NiTi) is called an exotic metal because it does not conform to the normal rules of metallurgy. NiTi alloy has special characteristics of superelasticity and shape memory. Superelasticity is associated with the occurrence of a phase transformation of the alloy upon the application of stress above a critical level, which takes place when the ambient temperature is above the so-called austenite-finish temperature of the material. This stress-induced martensitic transformation reverses spontaneously upon release of the stress; the material then returns to its original shape and size (2). This special property manifests as an enhanced elasticity of the NiTi alloy, allowing the material to recover after large strains. Thus, NiTi instruments appear highly flexible and elastic, hence the possibility of use in a continuous rotary fashion even in a curved canal. Despite these advantages, unexpected instrument fracture is not uncommon and represents a major concern in clinical use. Fatigue has been implicated to be the main reason for the fracture of endodontic files used clinically (2-5), with fatigue in the low-cycle region being most commonly implicated.

It is well known that the nature of the alloy and the manufacturing process greatly affect the instruments' mechanical behavior (6). To improve fracture resistance of NiTi files, manufacturers have either introduced new alloys to manufacture NiTi files or developed new manufacturing processes (7-10). Recently, NiTi rotary instruments made from a NiTi controlled memory wire (CM Wire; DS Dental, Johnson City, TN) have been introduced. The manufacturer claims that these instruments have flexibility and fatigue resistance superior to conventional NiTi rotary instruments made from superelastic wire. These new methods and materials for manufacturing NiTi instruments may advance the science of endodontic rotary instrumentation. However, these claims of the manufacturers have not been adequately tested by independent research.

A major drawback of most laboratory tests of the fatigue behavior of NiTi rotary instrument is that one has not been able to eliminate several confounding factors, such as material properties, design, and dimensions of the instrument, which are specific to the brand(s) being tested. This has made it difficult to quantify the effect of a single variable on fatigue behavior. The purpose of this study was to examine the fatigue behavior of NiTi instruments with two different designs made from a novel controlled memory NiTi alloy and conventional NiTi alloy. The ultimate goal of this research was to provide new insight about the rotary instruments made from CM Wire, which will lead to further improvement in their clinical performance.

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Materials and Methods

NiTi rotary instruments of .04 taper and size 25 ProFile (Dentsply Maillefer, Ballaigues, Switzerland), Typhoon (TYP), Typhoon CM (TYP CM), DS-SS0250425NEY (NEY), and DS-SS0250425NEY CM (NEY CM) (DS Dental, Johnson City, TN) were subjected to rotational bending at the curvature of 35° with an 8-mm radius and 45° with a 4.7-mm radius in air at the temperature of 23° ± 2°C. All tested instruments from DS dental company were prototypes, but according to the manufacturer they are identical with “Typhoon” instruments available soon commercially. Each group included 12 instruments. The fatigue testing protocol has been described previously and was reproduced throughout the experimental period (6, 11–14). Briefly, each NiTi instrument was constrained to a curvature by three rigid, stainless steel pins; a calibrated digital photograph was taken of the curvature. The instrument was then allowed to rotate at 300 rpm (as recommended by the manufactures) until fracture. The fatigue life, or total number of revolutions to failure, N_f, was recorded. Detached fragments were measured for length and then examined under scanning electron microscopy (Stereoscan 260; Cambridge Instruments, Cambridge, UK) operating at 5 to 8 kV; photomicrographs were taken of the fracture surface at various magnifications. For each instrument, the radius of curvature (R_c) at the site of fracture was determined on the photograph and the diameter of the fracture cross-section (d) on a photomicrograph by using computer software (ImageJ 1.34n; National Institutes of Health, Bethesda, MD). The maximum surface strain amplitude (ε_a) was then calculated as the ratio of the diameter of the fracture cross-section d (from a scanning electron photomicrograph) to two times the radius of the curvature of the instrument R_c (from a pretest calibrated photograph) (ε_a = d/2R_c) (11–14).

From the overall view, the crack initiation site(s) was identified by noting the chevron pattern, also called “herringbone marks” (15), on the fracture surface and by referring to the high-power views when necessary. The fractographic appearance of a fatigued metallic material always progresses from the crack origin to a zone of fatigue striations and, finally, a region of dimple rupture (16). The number of crack origin(s) for each specimen was recorded, and data were examined by using the Fisher exact test for the difference between different alloys and file designs. The region in which the dimple area could be found was outlined on the photomicrograph for each specimen, which was measured in software (ImageJ 1.34n) on each photomicrograph. Pearson correlation coefficients were calculated between N_f and surface strain amplitude, surface strain amplitude, and the area fraction occupied by the dimple region. The results were analyzed by using univariate analysis or post hoc analysis in software (SPSS for Windows 11.0; SPSS, Chicago, IL), when necessary, at a significance level of P < .05.

Results

A total of 120 NiTi rotary instruments were tested at 35° and 45° curvatures. The new alloy yielded an improvement of over three to eight times in N_f of CM files compared with conventional NiTi files (Table 1). The N_f life increased with decreasing strain amplitude. Both CM wire files (TYP CM and NEYY CM) had a significantly longer N_f and a lower surface strain amplitude than the conventional NiTi wire files with the same design (TYP and NEYY) at 35° and 45° (P < .05), whereas there was no significant difference on N_f among conventional NiTi wire files (ProFile, TYP, and NEYY) at both curvatures. Although there may be slight differences on N_f among instruments made from conventional NiTi wire (ProFile, TYP, and NEYY), the square (NEYY CM) versus triangular (TYP CM) configuration showed a significantly different lifetime on CM wire at both curvatures (P < .01). In general, instruments at the curvature of 35° were more resistant to fatigue failure compared with files of the same system at the curvature of 45° (P < .05). High coefficients of correlation were detected between the N_f and surface strain amplitude at 35° (r² = -0.79) and 45° (r² = -0.84) curvatures.

Fractographically, a single crack origin was usually found in conventional NiTi wire files (ProFile, TYP, and NEYY) at both curvatures (Fig. 1 and Table 2). CM wire instruments had a higher number of multiple crack origins than conventional NiTi wire files of the same design (Fisher exact test, P < .01), especially in NEYY CM instruments (92% files with multiple crack origins). Nearly all specimens showed fatigue crack initiation at one or more cutting edges of the fracture cross-section unless a subsurface void or inclusion was present elsewhere. An area of microscopic dimples was present on all fracture surfaces. Overall, there was a general trend of a rapidly declining life at large ratio of the dimple area to the whole area at both curvatures. A coefficient of correlation between the N_f and values of the area fraction occupied by the dimple region was found at 35° (r² = 0.67) and 45° (r² = 0.75).

Discussion

Several devices and methods have been used to investigate *in vitro* the cyclic fatigue fracture resistance of NiTi rotary endodontic instruments (8–11, 17). All of these studies attempt to simulate the rotation of the instrument within a curvature to determine how long it would last before fatigue fracture occurs. The rotating instrument is either confined in a glass or metal tube, in a grooved block-and-rod assembly, or in a sloped metal block or three-point bending pins. A three-point bending device is a rather new method to the endodontic literature although the principle has long been used in an engineering

TABLE 1. The Number of Revolutions to Fracture (N_f), the Maximum Surface Strain Amplitude (ε_a), and the Dimple Area/Total Cross-section Area on the Fractured Instrument (%) for Each Brand at the Curvature of 35° and 45° in Dry Condition

Size 25/.04	45°			35°		
	N _f	ε _a	Dimple area (%)	N _f	ε _a	Dimple area (%)
ProFile	486 ± 163	8.3 ± 1.2	71 ± 6	640 ± 180	6.2 ± 0.4	67 ± 8
TYP	376 ± 124	8.4 ± 0.5	75 ± 1	645 ± 231	6.0 ± 0.9	72 ± 5
TYP CM	1340 ± 160	4.2 ± 1.1	43 ± 17	2422 ± 1806	3.9 ± 0.7	28 ± 6
NEY	329 ± 92	6.4 ± 0.3	79 ± 3	1213 ± 430	3.6 ± 0.6	71 ± 4
NEY CM	2629 ± 125	3.4 ± 0.7	43 ± 9	3491 ± 1,782	2.2 ± 0.9	39 ± 6

There were significant differences on N_f (univariate analysis, P < .05), the maximum surface strain amplitude (univariate analysis, P < .05), and the dimple area/total cross-section area (univariate analysis, P < .05) of all instruments between at the curvature of 35° and 45°. There were significant differences on N_f (post hoc tests, P < .05), the maximum surface strain amplitude (post hoc tests, P < .05), and the dimple area/total cross-section area (post hoc tests, P < .01) between TYP and TYP CM files at the curvature of 35° and 45°. There were significant differences on N_f (post hoc tests, P < .01), the maximum surface strain amplitude (post hoc tests, P < .01) and the dimple area/total cross-section area (post hoc tests, P < .01) between NEYY and NEYY CM files at the curvature of 35° and 45°. There was a significant difference on N_f between TYP CM and DS NEYY CM files at the curvature of 35° and 45° (post hoc tests, P < .01).

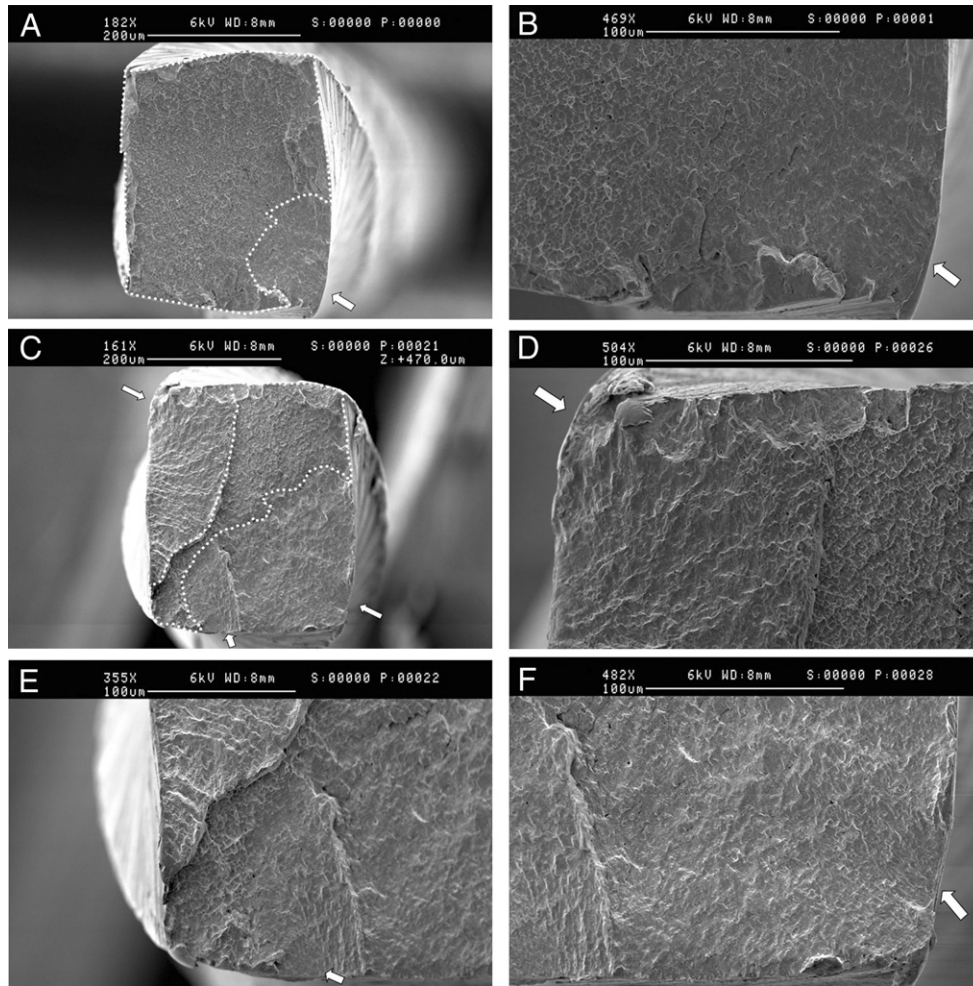


Figure 1. A photomicrograph of a fracture surface of NEYY files with the region of fatigue crack propagation and dimple area outlined (*dotted line*) with crack origins (*arrows*): (A) Overall view of NEYY file ($N_f = 380$; dimple area is 85%); (B) high magnification view of the crack origin (*arrow*); (C) overall view of DS NEYY CM file with three crack origins (*arrows*) ($N_f = 2390$; dimple area is 33%); (D) high magnification view of one crack origin (*arrow*); (E) high magnification view of the second crack origin (*arrow*); (F) high magnification view of the third crack origin (*arrow*).

context (18). It has been used to impose a curvature on the rotating instrument (6, 11–14, 19); the strain amplitude on the surface of the instrument may be estimated for each and every specimen with this method. The strain-life approach simulates the clinical situation; therefore, it is considered an appropriate means for examining the fatigue behavior of NiTi rotary files (6, 11–14). Most endodontic instrument manufacturers use NiTi raw wires of a very similar composition (Nitinol SE 508 or NiTi alloy with an Ni atomic percent of 50.8%).

ProFile instrument is one of the most tested NiTi rotary instruments manufactured from Nitinol SE 508. Therefore, the ProFile instrument was chosen as a gold standard made by conventional NiTi alloy. Only one instrument size of both brands (size #25) was tested because this is a commonly used size during instrumentation. The present study is the first study to compare rotary NiTi instruments manufactured to an identical file design but produced from two different sources of NiTi wires and from the same sources of NiTi

TABLE 2. Number of Crack Origins for Each Group in Dry Condition at Curvature of 35° and 45°

	45°		35°	
	1 crack origin	2 or more crack origins	1 crack origin	2 or more crack origins
ProFile	9	3	7	5
TYP	12	0	11	1
TYP CM	6	6	6	6
NEYY	11	1	9	3
NEYY CM	1	11	1	11

There were significant differences on the number of crack origins between TYP and TYP CM, NEYY and NEYY CM at the curvature of 45° (Fisher exact test, $P < .01$). There was a significant difference on the number of crack origins between NEYY and NEYY CM at the curvature of 35° (Fisher exact test, $P < .01$).

wire with two different designs, permitting direct study of the differences between alloys and file design.

The mechanical properties of NiTi alloy are easily influenced by small changes in composition, impurities, and heat treatment conditions (20). In particular, superelasticity and shape memory are strongly affected by heat treatment as part of the manufacturing processes (20–24). Recently, a proprietary thermomechanical processing procedure has been developed with the objective of producing superelastic NiTi wire blanks; it is termed M-Wire. The M-Wire technology allows the NiTi instruments more flexibility and resistance to cyclic fatigue compared with non-M-Wire NiTi instruments (7). Differential scanning calorimetric analyses found that at 37°C conventional superelastic NiTi wire has the austenite structure, whereas M-Wire is a mixture of nearly equal amounts of R-phase and austenite (25). Gao et al (10) reported that instruments made of M-Wire exhibited superior cyclic fatigue resistance compared with those made of regular superelastic wire. The results of the present study indicated that NiTi instruments made from CM Wire were nearly 300% to 800% more resistant to fatigue failure than instruments made from conventional NiTi wire. It is consistent with the view that the bulk material properties are the main determinant of fatigue life (14, 16). The relationship between structural modification and mechanical improvement requires, however, more rigorous investigations. More research is needed to identify different metallurgical characterization of CM Wire, M-Wire, and conventional NiTi wire.

Scanning electron microscopic observations of the fracture surfaces of the NiTi instruments indicated that fracture occurred according to the following model of fatigue failure (5, 11, 26): the presence of microscopic fatigue striations on all fracture surfaces, which were often located close to the crack origin and extended toward the centroid of the cross-section. Furthest away from the crack origin lay a region of “fast fracture” where features typical of dimple rupture, or so-called ductile failure (16, 26), could be observed. The crack origins were usually found at the cutting-edge region in most instruments. These data are in accordance with previous studies that found a similar mode that the first fatigue crack often is initiated at the cutting edge (14, 27).

The number of crack origin has been examined on discarded NiTi instruments after clinical use and laboratory tests, with instruments made from conventional NiTi wire (5, 6, 11–14, 27). The number of microcracks that form during fatigue depends on the stress or plastic strain amplitude. The fact is that the bulk material properties, ductility in particular, have a strong influence on the fatigue life behavior (28, 29). It is well known that the fatigue limit is not a definite fraction of the yield stress for all metals and alloys. Two additional factors are important: the degree of strain localization and the critical localized plastic strain to initiate the crack. The plastic strain amplitude may be made up by taking the two extremes of a small number of severe local plastic strains or a large number of small plastic strains. The latter case is much better for fatigue resistance, which benefits from any reduction in strain localization (26). Despite the significantly greater number of crack origins, the fatigue life was significantly higher on instruments made from CM Wire than from conventional wire, probably because of bulk mechanical properties and higher fatigue crack thresholds.

The local strain-life approach has gained acceptance as a useful method of evaluating the fatigue life of the metal. Our work focused on the study of the strain distribution of NiTi rotary instruments under two curvature conditions that may be encountered clinically. The results clearly indicated that as the curvature increases, a rapid increase in strain is to be expected. In this present study, instruments made from CM Wire (TYP CM and NEYY CM) had significantly higher N_f and lower surface

strain amplitude than the conventional NiTi wire files with an identical design in a dry condition. Cheung and Darvell (11) studied the relationship between the surface strain amplitude and fatigue life (ie, a strain-life relationship) on conventional NiTi instruments. In addition to the mean stress, many other factors can influence the strain-life fatigue behavior of a material. These include stress concentrations, residual stresses, multi-axial stress states, material properties, environmental effects, size, and surface finish (26). Further work is required to evaluate the strain-fatigue life curve (named the Coffin-Manson curve) of CM instruments.

The area occupied by the crack growth region (or dimple region) has not been previously examined quantitatively. Theoretically, the continuing reduction in the net area of the remaining intact section because of the progressive propagation of a fatigue crack would lower the load-bearing capacity of the part to such an extent that it fractures in the next load cycle as a result of simple overload. In the present study, the area occupied by the dimple region as a fraction of the total surface area of the fracture cross-section was measured for each specimen. A significant correlation between strain amplitude and the area fraction occupied by the dimple region was also discernible, which is in keeping with the study by Morgan et al (30). The values of the fraction area occupied by the dimple region were significantly smaller on NiTi instruments made from CM Wire than on instruments made from conventional NiTi wire. Hence, it is not surprising that CM series files had superior fatigue resistance than the other files made from conventional NiTi alloy.

It has been suggested that the cross-sectional configuration has little influence on the fatigue life of NiTi root canal instruments made from conventional NiTi wire (14). In the present study, there was no obvious difference on the fatigue life among ProFile, TYP (triangular configuration), and DS NEYY (square configuration) files, which were made from conventional NiTi wire. The result seemed to agree with earlier reported findings (14). Increasing the sample sizes would increase discriminating obscure small differences among groups. On the other hand, this is not supported by a study using numerical simulation, in which both the cross-section factor and material property had a substantial impact on fatigue lifetime (31). These findings lend support to our result in the present study; the square and triangular configuration of NiTi instruments made from CM Wire showed a significantly different fatigue life, probably related to the CM Wire properties. Therefore, the design of the instrument should also be taken into account because it is an important determinant of the fatigue lifetime.

The fatigue of NiTi alloys is sensitive to temperature, both locally and environmentally. On repeated loading, the latent heat of the stress-induced martensitic transformation released (and heat caused by work done) can elevate the local temperature, leading to a shortened fatigue life (32). The rotational-bending fatigue life of instruments in air used in this study standardized the working environment of each instrument although this model does not duplicate the *in vivo* situation. Further work is required to identify the fatigue life of different NiTi instruments in different solutions.

Within the limitations of this study, instruments made from CM Wire (TYP CM and NEYY CM) had significantly higher N_f and lower surface strain amplitude than the conventional NiTi wire files with the same design. CM Wire instruments gave a higher number of multiple crack origins than conventional NiTi wire files. The values of the area fraction occupied by the dimple region were significantly smaller on CM NiTi instruments than on conventional NiTi instruments.

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The authors deny any conflicts of interest related to this study.

References

1. Walia HM, Brantley WA, Gerstein H. An initial investigation of the bending and torsional properties of Nitinol root canal files. *J Endod* 1988;14:346–51.
2. Saburi T. Ti-Ni shape memory alloys. In: Otsuka K, Wayman CM, eds. *Shape Memory Materials*. Cambridge, UK: Cambridge University Press; 1998:49–96.
3. Sattapan B, Nervo GJ, Palamara JE, Messer HH. Defects in rotary nickel-titanium files after clinical use. *J Endod* 2000;26:161–5.
4. Shen Y, Cheung GS, Bian Z, Peng B. Comparison of defects in ProFile and ProTaper systems after clinical use. *J Endod* 2006;32:61–5.
5. Cheung GS, Peng B, Bian Z, Shen Y, Darvell BW. Defects in ProTaper S1 instruments after clinical use: fractographic examination. *Int Endod J* 2005;38:802–9.
6. Cheung GS, Darvell BW. Low-cycle fatigue of NiTi rotary instruments of various cross-sectional shapes. *Int Endod J* 2007;40:626–32.
7. Johnson E, Lloyd A, Kuttler S, Namerow K. Comparison between a novel nickel-titanium alloy and 508 nitinol on the cyclic fatigue life of ProFile 25/.04 rotary instruments. *J Endod* 2008;34:1406–9.
8. Larsen CM, Watanabe I, Glickman GN, He J. Cyclic fatigue analysis of a new generation of nickel titanium rotary instruments. *J Endod* 2009;35:401–3.
9. Kramkowski TR, Bahcall J. An in vitro comparison of torsional stress and cyclic fatigue resistance of ProFile GT and ProFile GT Series X rotary nickel-titanium files. *J Endod* 2009;35:404–7.
10. Gao Y, Shotton V, Wilkinson K, Phillips G, Johnson WB. Effects of raw material and rotational speed on the cyclic fatigue of ProFile Vortex rotary instruments. *J Endod* 2010;36:1205–9.
11. Cheung GS, Darvell BW. Fatigue testing of a NiTi rotary instrument: part 1-strainlife relationship. *Int Endod J* 2007;40:612–8.
12. Cheung GS, Shen Y, Darvell BW. Does electropolishing improve the low-cycle fatigue behavior of a nickel-titanium rotary instrument in hypochlorite? *J Endod* 2007;33:1217–21.
13. Cheung GS, Shen Y, Darvell BW. Effect of environment on low-cycle fatigue of a nickel-titanium instrument. *J Endod* 2007;33:1433–7.
14. Cheung GS, Darvell BW. Fatigue testing of a NiTi rotary instrument. Part 2: fractographic analysis. *Int Endod J* 2007;40:619–25.
15. Brooks CR, Choudhury A. *Failure analysis of engineering materials*. New York: McGraw-Hill; 2002.
16. Hull D. *Fractography: observing, measuring and interpreting fracture surface topography*. Cambridge, UK: Cambridge University Press; 1999.
17. Plotino G, Grande NM, Cordaro M, Testarelli L, Gambarini G. A review of cyclic fatigue testing of nickel-titanium rotary instruments. *J Endod* 2009;35:1469–76.
18. Eggeler G, Hornbogen E, Yawny A, Heckmann A, Wagner M. Structural and functional fatigue of NiTi shape memory alloys. *Mater Sci Eng A* 2004;378:24–33.
19. Zinelis S, Darabara M, Takase T, Ogane K, Papadimitriou GD. The effect of thermal treatment on the resistance of nickel–titanium rotary files in cyclic fatigue. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2007;103:843–7.
20. Condorelli GG, Bonaccorso A, Smecca E, Schäfer E, Cantatore G, Tripi TR. Improvement of the fatigue resistance of NiTi endodontic files by surface and bulk modifications. *Int Endod J* 2010;43:866–73.
21. Yoneyama T, Doi H, Hamanaka H, Yamamoto M, Kuroda T. Bending properties and transformation temperatures of heat treated Ni-Ti alloy wire for orthodontic appliances. *J Biomed Mater Res* 1993;27:399–402.
22. Thompson SA. An overview of nickel–titanium alloys used in dentistry. *Int Endod J* 2000;33:297–310.
23. Kuhn G, Tavernier B, Jordan L. Influence of structure on nickel–titanium endodontic instruments failure. *J Endod* 2001;27:516–20.
24. Kuhn G, Jordan L. Fatigue and mechanical properties of nickel–titanium endodontic instruments. *J Endod* 2002;28:716–20.
25. Alapati SB, Brantley WA, Iijima M, et al. Metallurgical characterization of a new nickel-titanium wire for rotary endodontic instruments. *J Endod* 2009;35:1589–93.
26. ASM International. *ASM handbook, vol. 19: fatigue and fracture*. Materials Park, OH: ASM International; 1996.
27. Shen Y, Cheung GS, Peng B, Haapasalo M. Defects in nickel-titanium instruments after clinical use. Part 2: fractographic analysis of fractured surface in a cohort study. *J Endod* 2009;35:133–6.
28. Reed-Hill RF, Abbaschian R. *Physical metallurgy principles*. 3rd edn. Boston, MA: PWS-Kent; 1992.
29. Schijve J. *Fatigue of structures and materials*. Dordrecht, The Netherlands: Kluwer Academic; 2001.
30. Morgan NB, Painter J, Moffat A. Mean strain effects and microstructural observations during in vitro fatigue testing of NiTi. In: SMST-2003. Proceedings of the International Conference on Shape Memory and Superelastic Technologies. Pacific Grove, CA: SMST Society; 2004:303–310.
31. Zhang EW, Cheung GSP, Zheng YF. A mathematical model for describing the mechanical behaviour of root canal instruments. *Int Endod* 2011;44:72–6.
32. Prymak O, Klocke A, Kahl-Nieke B, Epple M. Fatigue of orthodontic nickel-titanium (NiTi) wires in different fluids under constant mechanical stress. *Mater Sci Eng A* 2004;378:110–4.