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An overview of the mechanical properties of nickel–titanium endodontic instruments

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Differences in mechanical properties between endodontic instruments might be related to the chemical composition, phase constitution, or fabrication process of nickel–titanium (NiTi) endodontic instruments. An awareness and knowledge of the mechanical properties of NiTi endodontic files and their association with the metallurgical properties is useful for clinicians to understand the behavior of NiTi instruments in root canals and help them to make decisions regarding which instruments are appropriate for root canal therapy under certain clinical conditions. This review article summarizes the mechanical properties of NiTi endodontic instruments including the flexibility, torsional resistance, flexural resistance, and cyclic fatigue of the conventional superelastic NiTi instruments and the most recently developed novel NiTi instruments with martensite and R-phase. The influence of the metallurgical properties and the thermomechanical processing on the mechanical properties are discussed.

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Introduction

Nickel–titanium (NiTi) alloy has been used in endodontics for almost 25 years and has brought a major breakthrough to root canal therapy since Walia et al. (1) employed a nickel–titanium arch wire to fabricate a root canal file in 1988. NiTi endodontic instruments with superelasticity (SE) have gained extensive popularity amongst clinicians due to their higher flexibility and greater torsional resistance than the traditional stainless-steel ones (1,2). Therefore, an increasing number of superelastic NiTi endodontic files with various geometry designs (cross-sectional shape with or without "radial lands" or sharp cutting edges, constant or variable pitch, and progressive or constant taper) have been developed (3–5). However, the undesirable and unexpected separation of NiTi endodontic rotary files during root canal instrumentation caused by cyclic fatigue and/or torsional overload still remains a serious concern and drawback in clinical use. In recent years, novel kinds of NiTi endodontic files fabricated by proprietary thermomechanical processes such as M-wire files (e.g. ProFile GT Series X, ProFile Vortex, and ProFile Vortex Blue: Dentsply Tulsa Dental Specialties, Tulsa, OK, USA), controlled memory (CM) files (e.g. HyFlex CM: Coltene Whaledent, Cuyahoga Falls, OH and TYPHOON Infinite Flex NiTi: Clinician's Choice Dental Products, Milford, CT, USA), and R-phase wire [e.g. Twisted files (TF) and K3XF files: SybronEndo, Orange, CA] have been introduced and have shown improved flexibility and cyclic fatigue resistance compared to the traditional superelastic NiTi files (6–10).

The mechanical properties of NiTi endodontic instruments including flexibility, torsional resistance, and flexural fatigue are fundamental requirements of endodontic instruments for successful use. The flexibility is beneficial for maintaining the original shape of root canals, especially for the ones with severe curvatures. Adequate torsional resistance and flexural fatigue resistance favor reducing the occurrence of the intracanal separation. Thus, flexibility and resistance to fracture constitute properties expected for an ideal root canal file (11,12). From a material point of view, the properties of NiTi alloys depend on their chemical composition, phase constitution, and fabrication procedures, among which the metallurgical properties including the chemical composition and phase constitution are the internal factors and the fabrication procedures such as cold working, annealing, and aging are the external factors of the mechanical properties for NiTi endodontic files. Most research efforts to date have been devoted to the characterization of the metallurgical and mechanical properties of both the conventional superelastic NiTi instruments and the more recently developed novel NiTi instruments (13–17). In contrast, the relationship between the metallurgical properties and the mechanical properties of these instruments has been given relatively little attention. The knowledge of the mechanical properties and their association with the metallurgical properties of NiTi rotary instruments is helpful for clinicians to understand the behavior of NiTi instruments in root canals and to make decisions regarding which instruments are appropriate for root canal therapy. Therefore, this review is intended to summarize the mechanical properties of the traditional superelastic NiTi instruments and currently developed novel NiTi instruments, as well as their internal and external influencing factors, with a special focus on the influence of metallurgical properties on their mechanical properties.

Deformation mechanism of NiTi alloy

Nickel–titanium (NiTi) alloy is a kind of intermetallic compound with superior ductility, which is a stoichiometric compound of Ti and Ni. It is usually called NITINOL, where Ni is for Nickel, Ti is for Titanium, and NOL is for Naval Ordnance Laboratory, the place where Buehler and his co-workers discovered this alloy. It also called equiatomic NiTi alloy due to its one-to-one atomic ratio of nickel to titanium and referred to as NiTi alloy, respecting the metallurgical terminology of solid solutions with 55 wt% Ni and 45 wt% Ti (18). It is worth noting that only the NiTi alloys with nearly equiatomic ratio possess the unique superelasticity (SE) and shape memory effect (SME) because of the narrow solubility range of the "TiNi" phase at 500°C or below, which becomes negligible at about 500°C (19). That is why most of the NiTi alloys used for endodontic instruments are at a nearly one-to-one atomic ratio of nickel to titanium, which is equal to approximately 56 wt% nickel and 44 wt% titanium (2,7,20,21).

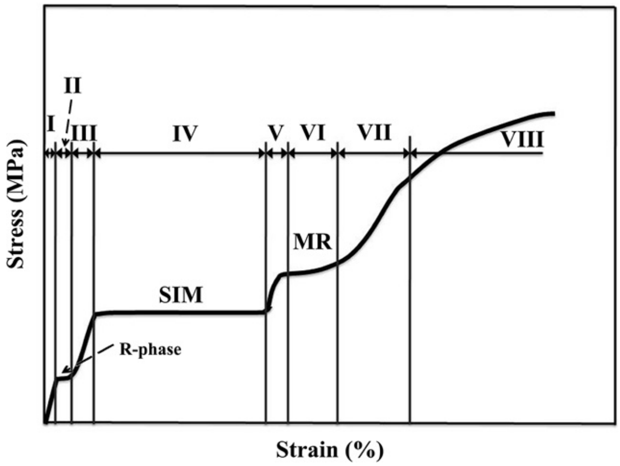

Fig. 1. Schematic drawing of tensile stress-strain curve of equiatomic NiTi alloy. It shows eight distinct stages: I, elastic deformation stage of austenite; II, stress plateau related to the transformation from austenite to R-phase; III, elastic deformation stage of R-phase induced by stress (stress-induced martensite, SIM); IV, stress plateau related to the transformation from R-phase to martensite caused by SIM; V, elastic deformation stage of martensite; VI, martensite reorientation (MR) stage; VII, uniform non-linear deformation stage of reoriented martensite; VIII, plastic deformation stage of reoriented martensite.

The illustrative tensile stress-strain curve of equiatomic NiTi alloy is shown in Figure 1. The mechanical behavior of near-equiatomic NiTi alloy exhibits eight stages and can be classified into three distinctive types according to the mechanism of its deformation, i.e. stress-induced martensitic (SIM) transformation (stage IV), martensite reorientation (MR, stage VI), and the distinct stage of yielding by plastic deformation (stage VIII) (22). The stress plateau in the stress-strain curve is an inelastic deformation associated with either SIM or MR, depending on the starting structure (e.g. deformed in austenitic state via SIM and deformed in martensitic state via MR) (23). The superelasticity is associated with SIM and the shape memory is associated with MR.

The superelasticity is one of the most important reasons for the use of NiTi alloys in endodontic instruments because it endows NiTi endodontic instruments with superior flexibility and allows them to follow the complex anatomy of root canals, resulting in reduced ledging and perforations (24). The superelasticity or pseudoelasticity (PE) of near-equiatomic NiTi alloy arises from the reversible stress-induced martensitic transformation. This process is driven by stress and influenced by the temperature difference between the working temperature and A_f, the critical finishing temperature for the reverse transformation of martensite on heating (25). A stress-induced martensitic transformation can be observed within the temperature range between M_s, the critical starting temperature for the forward transformation of austenite on cooling, and T_d, a critical temperature at which the critical stress for stress-induced martensitic transformation equals that of the plastic deformation of the austenite (26). The superelasticity of NiTi allows deformations of as much as 8% strain to be fully recoverable, in comparison with a maximum of less than 1% with other alloys such as stainless-steel (2). To utilize the superelasticity of NiTi alloys, it should be in the austenitic state, which is the high-temperature parent phase of NiTi alloy with B2 (CsCl) type ordered structure. Many conventional NiTi endondontic files were made of superelastic NiTi alloy, such as ProFile (Dentsply Maillefer, Ballaigues, Switzerland) and ProFile GT (Tulsa Dental Products, Tulsa, OK), Mtwo (VDW, Munich, Germany), LightSpeed (LightSpeed Technology, Inc., San Antonio, TX), Quantec (Analytic, Orange, CA), Hero 642 (Micro-Mega, Geneva, Switzerland), and K3 (SybronEndo, Orange, CA). All of these conventional rotary instruments are used clinically in the austenitic state (i.e. at body temperature). They can be inserted easily in the root canals due to the high elasticity and flexibility, and when an external stress is applied due to the torsion stress and the file friction against the canal inner walls, the stress-induced martensitic transformation occurs, giving rise to a more elastic material with a high ultimate tensile strength (27). In this way, the files work with a constant cutting stress, even in a much-curved canal. It is the reason why NiTi files are advantageous in maintaining the shape of root canals compared to stainless-steel ones.

Another mechanism of the mechanical deformation of NiTi alloys is the martensite variant reorientation (stage VI), which is related to the shape memory effect of NiTi alloys. When the NiTi alloy is deformed in the martensite state, it undergoes a strain, which is completely recoverable upon heating. This behavior is called the shape memory effect. Deformation via martensite reorientation may be observed at temperatures below A_s, the critical starting temperature for the reverse transformation of martensite on heating (26), and be completed at A_f. This process is thermally driven and influenced by the transformation temperatures. Peters et al. (6) pointed out that instruments in the martensitic phase can be easily deformed and will recover their shape when heated beyond the transformation temperature. The martensite phase of the NiTi alloy is a low-temperature phase with monoclinic structure (B19′), which possesses a relatively lower Yong's modulus (20 to 50 GPa) and yield strength (138 GPa) than the austenite (40 to 90 GPa and 379 MPa, respectively) (28–30). This indicated that the martensite is easily deformed at quite a low stress, whereas the austenite has much higher yield and flow stresses. Besides being more flexible than austenite, the martensite favors reducing the risk of file fracture under high stress because it can be plastically deformed rather than broken. Therefore, much effort has been devoted to introduce martensite into commercial NiTi endodontic instruments such as M-wire and CM endodontic instruments. The M-wire NiTi endodontic instruments (Dentsply Tulsa Dental Specialties) including ProFile GT-X, ProFile Vortex, and ProFile Vortex Blue are manufactured from the superelastic NiTi wires containing martensite. Several studies on the performance of the commercial instruments made with M-wire NiTi suggested enhanced flexibility and fatigue resistance (6–8,20). The CM files such as Hyflex CM file (Coltene Whaledent, Cuyahoga Falls, OH) and TYPHOON Infinite Flex NiTi files (Clinician's Choice Dental Products, Milford, CT, USA) are used in the martensitic state and possess the shape memory effect. These files can return to their original shape by autoclaving after use in curved canals. Moreover, the manufacturer of Hyflex CM files claims that their instruments are up to 300% more fatigue-resistant and have no rebound, and can regain their shape after sterilization. Some studies have confirmed the higher fatigue resistance of Hyflex CM files and Typhoon CM files when compared with conventional NiTi instruments (7,8,31,32).

In addition, there is a special type of martensitic transformation called R-phase transformation in NiTi alloys (stage II and III in Fig. 1). The R-phase is a rhombohedral phase that is incommensurate with the cubic B2 phase (33). The R-phase transformation occurs prior to the B2-B19′ transition and exhibited the characteristics of thermoelastic martensitic transformation, i.e. shape memory and superelasticity effects. Thus, the R-phase transformation can be temperature-induced and stress-induced in NiTi alloys. Compared with the martensite/austenite transformation in NiTi, the recoverable strain of the R-phase/austenite transformation (approximately 0.5%) is smaller and the temperature hysteresis is extremely small (1–2 K) (19). Moreover, the cyclic stability of the R-phase/austenite transformation is excellent (34) and the Young's modulus of the R-phase is typically lower than that of austenite. The appropriate choice of thermomechanical treatments can make the R-phase transformation occur due to the fact that the R-phase transformation tends to occur when precipitates or dislocations exist. The aging of Ni-rich NiTi alloy at the proper temperature may cause the precipitation of the Ti_3Ni_4 phase, and thermomechanical treatment (heat treatment after cold working) of NiTi alloy may create rearranged dislocation structures (19). Therefore, utilization of the R-phase transformation in the design and fabrication of NiTi endodontic instruments has the potential to produce new, innovative endodontic instruments. The TF file is a NiTi rotary instrument manufactured with the R-phase alloy using a twisting method; it exhibits a higher fatigue fracture resistance than ground files as reported in preliminary studies (35–40). K3XF is another product that takes advantage of R-phase technology and was developed by the same company as TF files (SybronEndo); however, it is fabricated by a grinding process rather than a twisting process. A special heat treatment is performed on K3XF files after the grinding process, which not only enhances the flexibility and strength, but also modifies the crystalline structure of the alloy to accommodate some of the internal stress caused by the grinding process (10,41). The manufacturer claims that K3XF files possess the basic features of the original K3 files plus an extraordinary new level of flexibility and resistance to cyclic fatigue owing to the proprietary R-phase technology. Recent studies (10,42,43) show that K3XF instruments have superior fatigue resistance compared with conventional superelastic NiTi instruments.

Relationship between the metallurgical properties and mechanical properties of NiTi endodontic instruments

The differences in the mechanical properties amongst NiTi endodontic instruments is due to the variety of materials and manufacturing conditions used, which affect their clinical performance (2,44–46). A NiTi alloy with a specific composition could have its mechanical properties optimized by adjusting the alloy's microstructure via cold work and heat treatment (47). Although the detailed thermomechanical process is unknown due to the protection of intellectual property, we can evaluate the influence of the thermomechanical treatments on the mechanical properties of NiTi endodontic instruments indirectly by analyzing the phase transformation behavior of the alloy. The characteristic transformation temperatures that can be manipulated by adjusting the chemical composition and heat treatment conditions play an important role in determining the alloy's mechanical properties (48). From the material point of view, the NiTi endodontic files have taken advantage of different phases in the NiTi alloy, i.e. conventional SE instruments, R-phase instruments, and martensite instruments. To a large extent, the differences in mechanical properties between NiTi endodontic files lie in the properties of the phases that exist in the alloy, which are determined by the phase transformation temperatures. Therefore, one can study the influence of phase constitution on the mechanical properties of NiTi endodontic files, instead of the influence of themomechanical processing. The typical metallurgical and mechanical properties of conventional NiTi and newly developed NiTi endodontic instruments are summarized in Table 1.

Metallurgical challenges in endodontic instruments

It is generally accepted that metallurgical properties have a dramatic impact on the performance of NiTi instruments. These properties include the composition, microstructure, and phase constitution of

Table 1: Typical metallurgical and mechanical properties of NiTi endodontic instruments

	SE-NiTi File			M-Wire File			CM-Wire File		R-Phase File	
Properties	ProFile	EndoSequence	K3	ProFile GTX	Vortex	Vortex Blue	Hyflex CM	Typhoon CM	TF File	K3XF
Ni content (wt%)	54.6[31]	56.0[31]	54.7[31] 54.2[42]	56[49]			52.1[31]			54.0[42]
A_f (°C)	31.1[16] 29.6 ± 2.4[50] 17.0[32]	31.1[32]	17.63 ± 1.76[42] 5.4 ± 6.8[50] 3.88 ± 3.21[51]		50.4[32] 51.7~55.9[52]		47~51.4[53]	55.1[32]	17.6[32] 18.9 ± 1.7[51]	24.89 ± 1.98[42]
Microhardness (MPa)	352.6 ± 10.5[54] 351 ± 11[31] 339 ± 29[55] 361 ± 39[46]	376 ± 18[42]	312 ± 18[42]	436 ± 10[49]	390 ± 7.9[54] 35.1 ± 27.6[52]	344.5 ± 7.6[54]	345 ± 16[31]			
Torque at fracture (g·cm)	100 ± 10[54] 61~201[56]	77~113[57]	7.29~25.03[42] 26~140[56]	31.2 ± 2[49] 66~168[56]	127 ± 9[54] 65~208[56] 109~146[57]	101 ± 7[7]	74~126[56] 47~138[6]		46~56.3[58] 47~61[57]	7.88~23.51[42]
Angle of rotation at fracture (°)	385 ± 32[54] 739.7 ± 55.7[45] 470~682[56]	336~346[57]	581.49~656.34[42] 1187~1412[56]	653 ± 78[49] 454~622[56]	367 ± 54[54] 415~772[56] 327~333[57]	457 ± 47[54]	562~822[56] 505~860[6]		609.0 ± 15.8[45] 538~541[57]	710.14~747.42[42]
Bending moment at 45 degrees (g·cm)	110 ± 8[54] 64.18 ± 4.2[7] 59.6 ± 5.06[35] 34~114[56]	48.98[59] 46.01 ± 3.9[7]	333 ± 39[51] 98.1 ± 6.4[10] 27~94[42] 163~406[42] 12~51[56]	31~90[56]	88 ± 4[54] 20~128[56] 59.06[59]	77 ± 6[54]	14.46 ± 2.7[7] 14~32[56] 35.6[59]		84 ± 14[51] 35.6 ± 2.89[35] 14.4~27.9 [58]	59.3 ± 4.3[10] 32~89[42] 123~280[42]
Fatigue number of cycles to failure (NCF)	638 ± 169[65] 352.6 ± 10.5[54] 369.4 ± 55.7[60]	205.9 ± 72.8[60]	552.5 ± 91.54[61] 579 ± 60.1[61] 439 ± 64[45] 542 ± 81[10]	801 ± 58[49] 762 ± 123[66] 570 ± 144.98[61] 561.5 ± 100.7[60]	1142 ± 179[54] 480 ± 104[43] 504.7 ± 104.2[60]	1609 ± 269[54]	2565 ~1572[6]	1340~ 2422[57]	769 ± 124[61] 731 ± 97[39] 769 ± 124[51] 506 ± 124[60]	651 ± 149[43] 1198 ± 279[10]

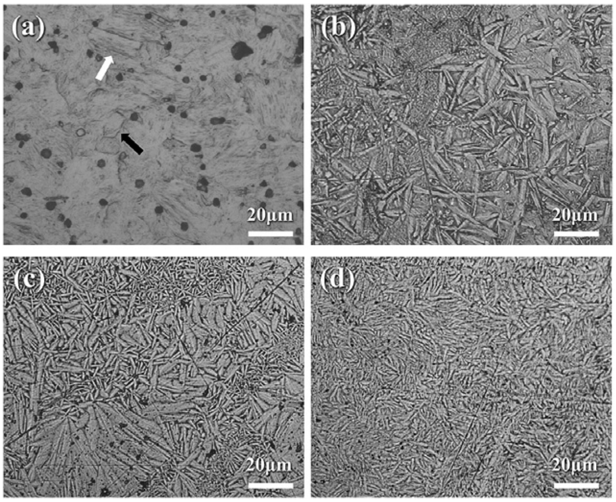

Fig. 2. Optical microscope images showing the microstructure of (a) TF file, (b) Vortex file, (c) Typhoon NiTi file, and (d) Typhoon CM file. The instruments were etched by mixed acid solution containing 3 mL of hydrofluoric acid, 5 mL of nitric acid, and 20 mL of acetic acid. The microstructure of the TF file shows both austenite (polygonal equiaxed grains, as shown by black arrow) and martensite (lath-like structure, as shown by white arrow) structures and the other three instruments show lenticular martensite structure.

NiTi instruments, and can be conveniently investigated by metallurgical laboratory techniques such as energy dispersive X-ray spectrometer (EDX), optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), normal differential scanning calorimeter (DSC), temperature-modulated DSC (TMDSC), and X-ray diffraction (XRD) spectrum or micro-XRD spectrum. These techniques are usually used together in order to form a more complete understanding of the metallurgical properties of NiTi instruments. For example, in the case of microstructure analysis, XRD spectrum can reveal the structure within approximately 50 μm of the surface (32), and DSC is usually used as a complimentary analytical technique that provides information on the phase transformation behavior. In addition, microstructural observation can further confirm the microstructure of the instrument. Figure 2 shows the typical microstructures of some NiTi instruments. The Typhoon NiTi file is a conventional NiTi instrument that has an austenitic microstructure. However, it shows lenticular martensitic microstructure under an optical microscope at room temperature. Shen et al. explained this phenomenon by the possible existence of stabilized martensite in the conventional superelastic NiTi instruments (32), which was confirmed by DSC results.

Conventional NiTi instruments take advantage of the superelasticity of NiTi alloy and are used in the austenite phase under clinical conditions. Recently, NiTi instruments containing martensite or R-phases have attracted the considerable attention of researchers due to the fact that they can be deformed more easily than those in the austenite phase. Therefore, proprietary thermomechanical processing procedures have been used in order to optimize the structure of the NiTi wire blanks for rotary instruments (36,40–42). The details of the metallurgical properties of newly developed NiTi instruments can be found in a previous review (41).

Influence of composition

Most of the NiTi alloys used for endodontic instruments are made in a nearly one-to-one atomic ratio of nickel to titanium, which equals approximately 56 wt% nickel and 44 wt% titanium (2,48,62). However, subtle adjustments in the ratio of these two elements cause a dramatic change in the transformation temperature, which has a significant effect on its mechanical properties (7). The nickel content of a NiTi alloy has a great influence on the transformation temperatures; an increase of 0.1% will lower the phase transformation temperature by 12°C (7). The chemical composition of NiTi endodontic files is usually examined by EDX, which is a semi-quantitative chemical analysis and has been employed for both conventional NiTi instruments and the most recently developed NiTi instruments (11,21,27,31,55,63). However, EDX is not sensitive enough to detect slight changes in the composition of the alloy that can result in a large difference in the mechanical properties (50). Zinelis et al. (31) evaluated the elemental composition of 10 brands of NiTi endodontic instruments and discovered that Hyflex X-files exhibit a lower percentage of nickel by weight (52 wt% Ni) than the common 54.5–57 wt% Ni shown in the great majority of commercially available NiTi rotary instruments. This lower nickel content exerts an influence on the transformation temperature, resulting in the A_f temperature of over 47°C for Hyflex NiTi files (53).

Influence of phase constitution

The phase constitution of NiTi endodontic files under the service temperature plays an important role in determining the mechanical properties of the files. The characteristic transformation temperatures and the crystalline phases found in the various instruments are an indication that different heat-treatment procedures may have been applied during instrument manufacture (48). The transformation temperatures (M_s, M_f, A_s, and A_f) at which the crystal structure changes from austenite to martensite or vice versa are usually measured by DSC. If the temperature is above A_f, the NiTi alloy is fully austenite, which possesses superior superelasticity. If the temperature is below M_f, the alloy possesses a shape memory effect due to the phase constitution of fully martensite. The presence of some martensite facilitates the stress-induced martensitic transformation, which occurs at lower applied stresses. Thus, less stress is necessary to induce martensitic transformation at a higher M_s (45). Viana et al. (48) revealed that K3 instruments with a lower A_f temperature showed a higher bending moment due to the small amounts of martensite. If the temperature is above A_s and below A_f, or above M_f and below M_s, the alloy consists of austenite and martensite. In addition, DSC curves can provide more information about the phase transformation behavior upon cooling and heating such as one-stage phase transformation (B2-B19′) and multi-stage phase transformation (B2-R-B19′), which can further reveal the influence of the thermomechanical process. However, the results of DSC analyses in NiTi endodontic instruments are contradictory. As Bahia et al. (21) pointed out, the A_s temperature of unused ProFile instruments tested by Brantley et al. (13) was -33°C, which certainly should not be expected for NiTi superelastic alloys (64,65). This may be attributed to the unique form of NiTi endodontic files with complex flutes and small diameters. Therefore, other techniques are needed to confirm the results of DSC, such as XRD (13–17), micro-XRD (66), metallographic examination (17), and TEM (9,66). However, there are some limitations of each technique. Only the phase with an amount more than 3% in volume can be detected by XRD due to the restriction of the resolution of the technique. Miyai et al. (50) analyzed the phase constitution of similar instruments using DSC experiments and found that instruments with low transformation temperatures (Hero, K3) tended to show a higher maximum torque and higher bending load value than instruments with high transformation temperatures (EndoWave, ProFile, and ProTaper). They indicated that the mechanical performance of NiTi endodontic instruments may be closely correlated with the transformation behavior of the alloy.

Mechanical performance of NiTi endodontic instruments

Flexibility

Flexibility of endodontic instruments is a fundamental requirement (48) because it allows appropriate canal enlargement while maintaining the instrument centered within the canal (67), causing fewer undesirable changes in the shape of curved canals (11). The high flexibility of NiTi endodontic instruments comes from the combination of the low elastic modulus and unique superelastic properties (48,68), which means that NiTi files can undergo significant deformation without reaching their elastic limits and still return to their original form. Flexibility is influenced by chemical composition and heat treatment as well as geometric configuration (48). In laboratory studies, it is usually evaluated by the bending test because bending is the most representative loading caused by the curvature of the root canals (68). The flexibility is characterized by the bending moment at 45° using a testing apparatus built according to ISO 3630-1 specifications (77) or studied by load-deflection curves during the loading and unloading process using a cantilever bending apparatus, as shown in Figure 3. Low bending moment and high flexural stiffness is indicative of the high flexibility.

For conventional SE NiTi endodontic instruments, the influence of geometrical designs play a crucial role on the flexibility, such as the cross-sectional configuration, the depth of the flute, area of the inner core, and peripheral mass in cross-section (69). Viana et al. (48) found a linear relationship between the average bending moment and the instrument's diameter and cross-sectional area at 3 mm from the tip for ProTaper Universal, K3, and EndoSequence instruments. It has also been reported by previous studies that the U-file design had the lowest flexural rigidity, compared with a "convex triangular" cross-section with or without an additional flute (69). A

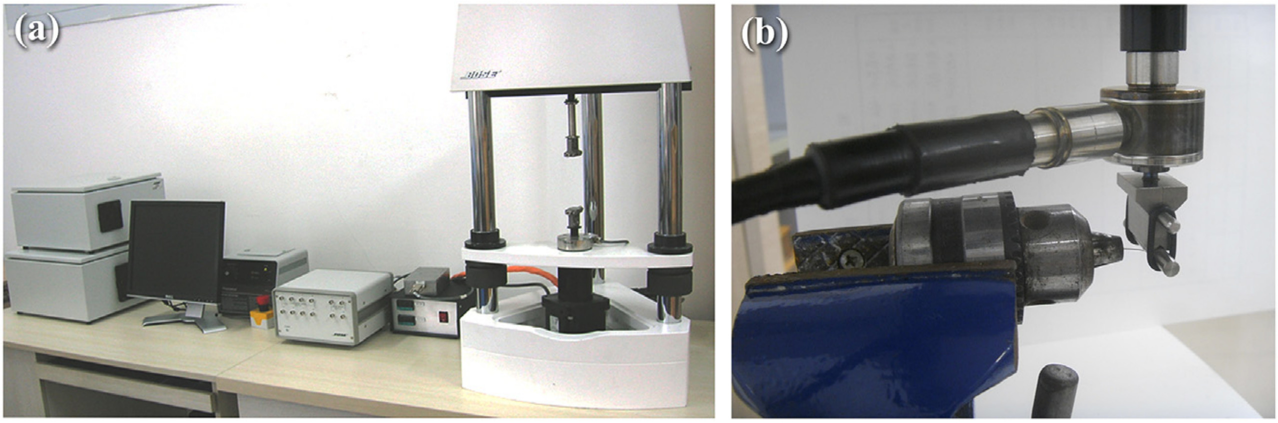

Fig. 3. The apparatus for the cantilever bending test. (a) Biomechanical performance test system (ElectroForce 2300, Bose Corporation ElectroForce Systems Group). (b) Set-up of the cantilever bending test.

recent numerical analysis concerning the effect of pitch and cross-sectional characteristics indicated that the flexural stiffness is related to the center-core area and that a decrease in pitch will lower the flexural stiffness (70). However, for the newly developed thermomechanically treated NiTi endodontic instruments, flexibility is largely determined by the manufacturing processes. Hayashi et al. (45) found that the additional heat treatment of hybrid NiTi instruments was effective in increasing the flexibility of NiTi rotary instruments. As demonstrated by a previous study, the new manufacturing process possesses the capability to produce more flexible NiTi instruments (TF files) (35).

Torsional resistance

Torsion is one of the primary mechanisms responsible for the intracanal separation of NiTi endodontic instruments (71,72), which accounts for 55.7% of the failures of NiTi rotary instruments (71,72). It can be generated within the rotary file when the tip or some part of the instrument is locked against the canal wall while the shank of the file (driven by the handpiece) continues to rotate or is subjected to excessive pressure by the clinician (3,68). The tip fractures when the handpiece torque exceeds the ultimate strength of the metal (73). High torsional stiffness is desirable for the clinical performance of rotary files owing to the enhanced cutting efficiency and reduced torsional failure risk (11,74,75). In addition, the amount of rotation an endodontic file can withstand prior to failure reflects its ductility, which can be considered as a "safety factor" for endodontic files. It is easier to detect a deformation of the cutting spiral after having used a file with greater ductility; this helps to prevent failure of the file by allowing it to be discarded prior to breakage (76). The measurement of the torsional strength of root canal instruments is usually performed in a torsion meter according to the method proposed by ISO3630-1 (77) or ANSI/ADA Specification No. 28 for stainless-steel root canal reamers and files (41,77,78). The typical torque-angle curves are shown in Figure 4. As can be seen in the figures, two parameters (i.e. the maximum torque and angular distortion at the fracture of the instrument) are obtained from the test (42,74). The torsional resistance of a NiTi endodontic file is affected by material properties, manufacturing quality, and geometric design. Several studies have found that some thermomechanically treated NiTi instruments show similar torque at the fracture to that of conventional SE NiTi instruments (6,79,80). In contrast, the results of a recent study showed that Vortex Blue NiTi and M-wire offered functional advantages over conventional superelastic NiTi, and Vortex Blue showed improved fatigue resistance and flexibility compared with ProFile Vortex M-wire (54).

Flexural fatigue resistance

Cyclic fatigue occurs when a metal is subjected to repeated cycles of tension and compression that cause its structure to break down, ultimately leading to fracture (600). It is the main reason for the majority (93%) of broken instruments. The fracture caused by

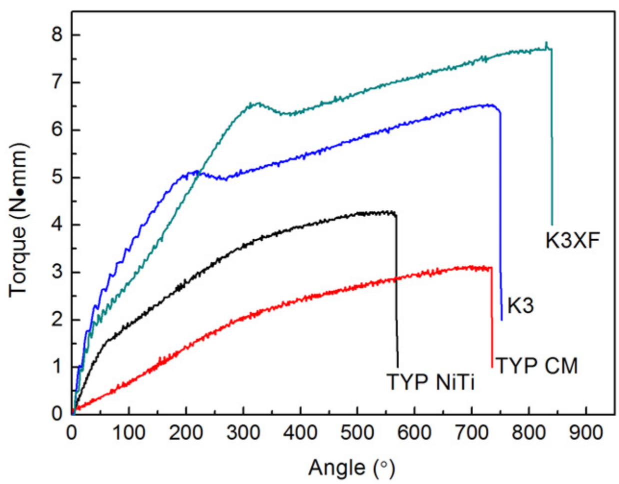

Fig. 4. Typical torque-angle curves of four NiTi endodontic instruments evaluated by the torsional test: Typhoon NiTi (TYP NiTi; DS Dental, Johnson City, TN), Typhoon CM (TYP CM; DS Dental, Johnson City, TN), K3 (SybronEndo, Orange, CA), and K3XF (SybronEndo). All instruments are size 25/0.04.

cyclic fatigue of NiTi endodontic instruments is difficult to detect during clinical practice due to the invisible signs of permanent deformation during cyclic fatigue (81). That is why many attempts have been made to improve the resistance to cyclic fatigue for NiTi instruments, including novel thermomechanical processing and exploring new materials. The cyclic fatigue properties of NiTi endodontic instruments have been studied fairly extensively (36,38,44,82–96). The fatigue life is determined by two factors: the rate of crack initiation and the rate of crack propagation. It has been stated that M-wire instruments demonstrate superior resistance to fatigue-crack initiation compared with regular SE-wire files, owing to the better reorientation capability of the M variants (83). In addition, a hybrid microstructure with a certain proportion of martensite is more resistant to crack propagation than a fully austenitic microstructure due to the fact that the speed of fatigue crack propagation of austenite is much faster than martensite (83). However, there is not yet any specification or international standard to test the cyclic fatigue resistance of endodontic rotary instruments. The devices and methods used to investigate the *in vitro* cyclic fatigue properties of NiTi rotary endodontic instruments are based on various simulated root canals (artificial root canals) with different curvatures, such as a glass or metal tube, a grooved block-and-rod assembly, and a sloped metal block (91). It is worth noting that even a small variation in the parameters of the curvature followed by an instrument subjected to flexural fatigue may result in a significant influence on the results of the cyclic fatigue (91,92). Most of the artificial root canal models used for evaluating *in vitro* cyclic fatigue resistance have the drawback of the difference between the real trajectory of a NiTi instrument inside the artificial root canals and the predetermined curvatures, making it impossible to compare the cyclic fatigue between instruments of different sizes under different conditions. Plotino et al. (91–94) proposed a new model specifically constructed on the dimensions of the instrument tested to reduce the approximation in cyclic fatigue tests, which were made using a die-sinking electrical-discharge machining process. It has been stated that different NiTi rotary instruments may follow a precise and repeatable trajectory in terms of radius and angle of curvature (92).

Mechanical properties under combined loading modes

NiTi endodontic instruments used in clinical practice are subjected to both flexural fatigue and torsional load simultaneously during root canal preparation procedures, probably leading to instrument separation due to hybrid forces (97). Because bending is imposed by the root canal anatomy, in the case of blockage, it is also associated with torsion (68). Clinically, both cyclic fatigue and torsional failure probably occur simultaneously; however, most studies on the fracture simulation of NiTi files have been performed separately for cyclic fatigue or torsional failure (74,80,89). Several researchers have attempted to evaluate the effect of combined cyclic fatigue and torsional stress on the performance of NiTi rotary instruments (89,98–101). It has been reported that cyclic fatigue of approximate 75% may significantly reduce the torsional resistance of NiTi rotary instruments (8). Ulmann & Peters (100) found that cyclic pre-stressing significantly reduced torsional resistance in ProTaper finishing files, while ProTaper shaping files were largely unaffected. In a recent study, it was found that the combined stress of cyclic fatigue and torsional load compared to cyclic fatigue only resulted in statistically significantly different mean fragment lengths, independent of file diameter, alloy, or cross-section. It suggested that stress was distributed from areas where torsional load was applied toward the area undergoing cyclic fatigue and implied that clinically a fracture resulting from only cyclic fatigue may not be distinguished from a fracture due to the combined stress from cyclic fatigue and torsional stress (101).

Conclusions

The differences in the mechanical properties amongst NiTi endodontic files depend mostly upon the properties of the phases that exist in the alloy, which is determined by the phase transformation temperature. Although the detailed thermomechanical process is unknown due to the protection of intellectual property, one can evaluate the influence of thermomechanical treatments on the mechanical properties of NiTi endodontic instruments indirectly by analyzing the phase transformation behavior of the alloy. From the material point of view, NiTi endodontic files have taken advantage of the different phases in NiTi alloy, i.e. conventional SE instruments, R-phase instruments, and martensite instruments. Compared with SE NiTi instruments, the introduction of martensite and R-phases into the phase constitution of recently developed thermomechanically treated NiTi endodontic instruments has produced improved flexibility and fracture resistance.

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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**: 346–351.
2. Thompson SA. An overview of nickel–titanium alloys used in dentistry. *Int Endod J* 2000: **33**: 297–310.
3. Arbab-Chirani R, Chevalier V, Arbab-Chirani S, Calloch S. Comparative analysis of torsional and bending behavior through finite-element models of 5 NiTi endodontic instruments. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2011: **111**: 115–121.
4. Hülsmann M, Peters OA, Dummer PMH. Mechanical preparation of root canals: shaping goals, techniques and means. *Endod Topics* 2005: **10**: 30–76.
5. Diemer F, Calas P. Effect of pitch length on the behavior of rotary triple helix root canal instruments. *J Endod* 2004: **30**: 716–718.
6. Peters OA, Gluskin AK, Weiss RA, Han JT. An *in vitro* assessment of the physical properties of novel Hyflex nickel–titanium rotary instruments. *Int Endod J* 2012: **45**: 1027–1034.
7. Testarelli L, Plotino G, Al-Sudani D, Vincenzi V, Giansiracusa A, Grande NM, Gambarini G. Bending properties of a new nickel–titanium alloy with a lower percent by weight of nickel. *J Endod* 2011: **37**: 1293–1295.
8. Shen Y, Qian W, Abtin H, Gao Y, Haapasalo M. Fatigue testing of controlled memory wire nickel–titanium rotary instruments. *J Endod* 2011: **37**: 997–1001.
9. Ye J, Gao Y. Metallurgical characterization of M-wire nickel–titanium shape memory alloy used for endodontic rotary instruments during low-cycle fatigue. *J Endod* 2012: **38**: 105–107.
10. Gambarini G, Plotino G, Grande NM, Al-Sudani D, De Luca M, Testarelli L. Mechanical properties of nickel–titanium rotary instruments produced with a new manufacturing technique. *Int Endod J* 2011: **44**: 337–341.
11. Schäfer E, Dzepina A, Danesh G. Bending properties of rotary nickel–titanium instruments. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2003: **96**: 757–763.
12. Yared G, Kulkarni GK, Ghossayn F. An *in vitro* study of the torsional properties of new and used K3 instruments. *Int Endod J* 2003: **36**: 764–769.
13. Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH. Differential scanning calorimetric studies of nickel–titanium rotary endodontic instruments after simulated clinical use. *J Endod* 2002: **28**: 774–778.
14. Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH. Differential scanning calorimetric studies of nickel–titanium rotary endodontic instruments. *J Endod* 2002: **28**: 567–572.
15. Brantley WA, Iijima M, Grentzer TH. Temperature-modulated DSC provides new insight about nickel–titanium wire transformations. *Am J Orthod Dentofacial Orthop* 2003: **124**: 387–394.
16. Alexandrou GB, Chrissafis K, Vasiliadis LP, Pavlidou E, Polychroniadis EK. SEM observations and differential scanning calorimetric studies of new and sterilized nickel–titanium rotary endodontic instruments. *J Endod* 2006: **32**: 675–679.
17. Alapati SB, Brantley WA, Iijima M, Schricker SR, Nusstein JM, Li UM, Svec TA. Micro-XRD and temperature-modulated DSC investigation of nickel–

titanium rotary endodontic instruments. *Dent Mater* 2009: **25**: 1221–1229.
18. Filip P. Titanium–nickel shape memory alloys in medical applications. In: Brunette DM, Tengvall P, Textor M, Thomsen P, eds. *Titanium in Medicine: Material Science, Surface Science, Engineering, Biological Responses and Medical Applications.* Berlin, Germany: Heidelberg, 2001: 54–80.
19. Otsuka K, Ren X. Physical metallurgy of Ti–Ni based shape memory alloys. *Prog Mater Sci* 2005: **50**: 511–678.
20. Shen Y, Qian W, Abtin H, Gao Y, Haapasalo M. Effect of environment on fatigue failure of controlled memory wire nickel–titanium rotary instruments. *J Endod* 2012: **38**: 376–380.
21. Bahia MGA, Martins RC, Gonzalez BM, Buono VTL. Physical and mechanical characterization and the influence of cyclic loading on the behaviour of nickel–titanium wires employed in the manufacture of rotary endodontic instruments. *Int Endod J* 2005: **38**: 795–801.
22. Tan G, Liu Y. Comparative study of deformation-induced martensite stabilisation via martensite reorientation and stress-induced martensitic transformation in NiTi. *Intermetallics* 2004: **12**: 373–381.
23. Zhou HM, Shen Y, Zheng W, Li L, Zheng YF, Haapasalo M. Mechanical properties of controlled memory and superelastic nickel–titanium wires used in the manufacture of rotary endodontic instruments. *J Endod* 2012: **38**: 1535–1540.
24. Kenneth FL. Endodontic instruments for root canal therapy. *Clin Tech Small Anim Pract* 2001: **16**: 139–150.
25. McCormick PG, Liu Y, Miyazaki S. Intrinsic thermal-mechanical behaviour associated with the stress-induced martensitic transformation in NiTi. *Mater Sci Eng A* 1993: **167**: 51–56.
26. Liu YN, Xiang H. Apparent modulus of elasticity of near-equiatomic NiTi. *J Alloys Compd* 1998: **270**: 154–159.
27. Torrisi L. The NiTi superelastic alloy application to the dentistry field. *Biomed Mater Eng* 1999: **9**: 39–47.
28. Zadno R, Duerig TW. Linear superelasticity in cold-worked NiTi. In: Duerig TW, Melton KN, Stockel D, Wayman CM, eds. *Engineering Aspects of Shape Memory Alloys.* London: Butterworth-Heinemann, 1990: 414.
29. Bidaux JE, Bataillard L, Manson JA, Gotthardt R. Phase transformation behavior of thin shape memory alloy wires embedded in a polymer matrix composite. *Proc 3rd Eur Conf on Advanced Materials and Processes* 1993: 1–5.
30. Hodson DE, Wu MH, Biermann RJ. Shape memory alloys. In: *Metals Handbook*, 10th edn. ASM International, 1990: **2**: 897–902.
31. Zinelis S, Eliades T, Eliades G. A metallurgical characterization of ten endodontic NiTi instruments: assessing the clinical relevance of shape memory and superelastic properties of NiTi endodontic instruments. *Int Endod J* 2010: **43**: 125–134.
32. Shen Y, Zhou HM, Zheng YF, Campbell L, Peng B, Haapasalo M. Metallurgical characterization of controlled memory wire nickel–titanium rotary instruments. *J Endod* 2011: **37**: 1566–1571.
33. Zhang XY, Sehitoglu H. Crystallography of the B2→R→B19′ phase transformations in NiTi. *Mater Sci Eng A* 2004: **374**: 292–302.
34. Tobushi H, Yamada S, Hachisuka T, Ikai A, Tanaka K. Cyclic deformation properties in a wire of TiNi shape memory alloy subjected to cyclic loadings. In: Abe T, Tsuta T, eds. *Advances in Engineering Plasticity and its Applications.* Oxford, UK: Pergamon Press, 1996: 611–616.
35. Gambarini G, Gerosa R, De Luca M, Garala M, Testarelli L. Mechanical properties of a new and improved nickel–titanium alloy for endodontic use: an evaluation of file flexibility. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2008: **105**: 798–800.
36. Larsen CM, Watanabe I, Glickman GN, He J. Cyclic fatigue analysis of a new generation of nickel–titanium rotary instruments. *J Endod* 2009: **35**: 401–403.
37. Bhagabati N, Yadav S, Talwar S. An *in vitro* cyclic fatigue analysis of different endodontic nickel–titanium rotary instruments. *J Endod* 2012: **38**: 515–518.
38. Rodrigues RC, Lopes HP, Elias CN, Amaral G, Vieira VT, De Martin AS. Influence of different manufacturing methods on the cyclic fatigue of rotary nickel–titanium endodontic instruments. *J Endod* 2011: **37**: 1553–1557.
39. Kim HC, Yum J, Hur B, Cheung GS. Cyclic fatigue and fracture characteristics of ground and twisted nickel–titanium rotary files. *J Endod* 2010: **36**: 147–152.
40. Ha JH, Kim SK, Cohenca N, Kim HC. Effect of R-phase heat treatment on torsional resistance and cyclic fatigue fracture. *J Endod* 2013: **39**: 389–393.
41. Shen Y, Zhou HM, Zheng YF, Peng B, Haapasalo M. Current challenges and concepts of the thermomechanical treatment of nickel–titanium instruments. *J Endod* 2013: **39**: 163–172.
42. Shen Y, Zhou HM, Wang Z, Campbell L, Zheng YF, Haapasalo M. Phase transformation behavior and mechanical properties of thermomechanically treated K3XF nickel–titanium instruments. *J Endod* 2013: **39**: 919–923.
43. Plotino G, Costanzo A, Grande NM, Petrovic R, Testarelli L, Gambarini G. Experimental evaluation on the influence of autoclave sterilization on the cyclic fatigue of new nickel–titanium rotary instruments. *J Endod* 2012: **38**: 222–225.
44. 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**: 843–847.
45. Hayashi Y, Yoneyama T, Yahata Y, Miyai K, Doi H, Hanawa T, Ebihara A, Suda H. Phase transformation

behaviour and bending properties of hybrid nickel–titanium rotary endodontic instruments. *Int Endod J* 2007: **40**: 247–253.
46. Viana AC, Gonzalez BM, Buono VT, Bahia MG. Influence of sterilization on mechanical properties and fatigue resistance of nickel–titanium rotary endodontic instruments. *Int Endod J* 2006: **39**: 709–715.
47. Pelton AR, Dicello J, Miyazaki S. Optimization of processing and properties of medical-grade Nitinol wire. In: Russell S, Pelton A, eds. *SMST. Proceedings of the International Conference on Shape Memory and Superelastic Technologies.* Pacific Grove, 2000.
48. Viana AC, Chaves Craveiro de Melo M, Guiomar de Azevedo Bahia M, Lopes Buono VT. Relationship between flexibility and physical, chemical, and geometric characteristics of rotary nickel–titanium instruments. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2010: **110**: 527–533.
49. da Cunha Peixoto IF, Pereira ES, da Silva JG, Viana AC, Buono VT, Bahia MG. Flexural fatigue and torsional resistance of ProFile GT and ProFile GT series X instruments. *J Endod* 2010: **36**: 741–744.
50. Miyai K, Ebihara A, Hayashi Y, Doi H, Suda H, Yoneyama T. Influence of phase transformation on the torsional and bending properties of nickel–titanium rotary endodontic instruments. *Int Endod J* 2006: **39**: 119–126.
51. Hou X, Yahata Y, Hayashi Y, Ebihara A, Hanawa T, Suda H. Phase transformation behaviour and bending property of twisted nickel–titanium endodontic instruments. *Int Endod J* 2011: **44**: 253–258.
52. Shen Y, Coil JM, Zhou HM, Tam E, Zheng YF, Haapasalo M. ProFile Vortex instruments after clinical use: a metallurgical properties study. *J Endod* 2012: **38**: 1613–1617.
53. Shen Y, Coil JM, Zhou H, Zheng Y, Haapasalo M. HyFlex nickel–titanium rotary instruments after clinical use: metallurgical properties. *Int Endod J* 2013: **46**: 720–729.
54. Gao Y, Gutmann JL, Wilkinson K, Maxwell R, Ammon D. Evaluation of the impact of raw materials on the fatigue and mechanical properties of ProFile Vortex rotary instruments. *J Endod* 2012: **38**: 398–401.
55. Kim JW, Griggs JA, Regan JD, Ellis RA, Cai Z. Effect of cryogenic treatment on nickel–titanium endodontic instruments. *Int Endod J* 2005: **38**: 364–371.
56. Ninan E, Berzins DW. Torsion and bending properties of shape memory and superelastic nickel–titanium rotary instruments. *J Endod* 2013: **39**: 101–104.
57. Wycoff RC, Berzins DW. An *in vitro* comparison of torsional stress properties of three different rotary nickel–titanium files with a similar cross-sectional design. *J Endod* 2012: **38**: 1118–1120.
58. Braga LC, Magalhães RR, Nakagawa RK, Puente CG, Buono VT, Bahia MG. Physical and mechanical properties of twisted or ground nickel–titanium instruments. *Int Endod J* 2013: **46**: 458–465.
59. Pongione G, Pompa G, Milana V, Di Carlo S, Giansiracusa A, Nicolini E, De Angelis F. Flexibility and resistance to cyclic fatigue of endodontic instruments made with different nickel–titanium alloys: a comparative test. *Ann Stomatol (Roma)* 2012: **3**: 119–122.
60. Hibbeler RC. *Mechanics of Materials,* 3rd edn. Upper Saddle River, NJ: Prentice Hall, 1997.
61. Gambarini G, Grande NM, Plotino G, Somma F, Garala M, De Luca M, Testarelli L. Fatigue resistance of engine-driven rotary nickel–titanium instruments produced by new manufacturing methods. *J Endod* 2008: **34**: 1003–1005.
62. Ounsi HF, Al-Shalan T, Salameh Z, Grandini S, Ferrari M. Quantitative and qualitative elemental analysis of different nickel–titanium rotary instruments by using scanning electron microscopy and energy dispersive spectroscopy. *J Endod* 2008: **34**: 53–55.
63. Bonaccorso A, Schäfer E, Condorelli GG, Cantatore G, Tripi TR. Chemical analysis of nickel–titanium rotary instruments with and without electropolishing after cleaning procedures with sodium hypochlorite. *J Endod* 2008: **34**: 1391–1395.
64. Otsuka K, Wayman CM. *Shape Memory Materials*, 1st edn. Cambridge, UK: Cambridge University Press, 1998.
65. Liu Y, Xie ZL, Van Humbeeck J. Cyclic deformation of NiTi shape memory alloys. *Mater Sci Eng A Struct Mater* 1999: **273–275**: 673–678.
66. Alapati SB, Brantley WA, Iijima M, Clark WA, Kovarik L, Buie C, Liu J, Ben Johnson W. Metallurgical characterization of a new nickel–titanium wire for rotary endodontic instruments. *J Endod* 2009: **35**: 1589–1593.
67. Tepel J, Schäfer E. Endodontic hand instruments: cutting efficiency, instrumentation of curved canals, bending and torsional properties. *Endod Dent Traumatol* 1997: **13**: 201–210.
68. Chevalier V, Arbab-Chirani R, Arbab-Chirani S, Calloch S. An improved model of 3-dimensional finite element analysis of mechanical behavior of endodontic instruments. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2010: **109**: 111–121.
69. Kim TO, Cheung GS, Lee JM, Kim BM, Hur B, Kim HC. Stress distribution of three NiTi rotary files under bending and torsional conditions using a mathematic analysis. *Int Endod J* 2009: **42**: 14–21.
70. Versluis A, Kim HC, Lee W, Kim BM, Lee CJ. Flexural stiffness and stresses in nickel–titanium rotary files for various pitch and cross-sectional geometries. *J Endod* 2012: **38**: 1399–1403.
71. Alapati SB, Brantley WA, Svec TA, Powers JM, Nusstein JM, Daehn GS. SEM observations of nickel–titanium rotary endodontic instruments that fractured during clinical use. *J Endod* 2005: **31**: 40–43.
72. Sattapan B, Palamara JE, Messer HH. Torque during canal instrumentation using rotary nickel–titanium files. *J Endod* 2000: **26**: 156–160.
73. Martín B, Zelada G, Varela P, Bahillo JG, Magán F, Ahn S, Rodríguez C. Factors influencing the fracture

of nickel–titanium rotary instruments. *Int Endod J* 2003: **36**: 262–266.

74. Yum J, Cheung GS, Park JK, Hur B, Kim HC. Torsional strength and toughness of nickel–titanium rotary files. *J Endod* 2011: **37**: 382–386.
75. Berutti E, Chiandussi G, Gaviglio I, Ibba A. Comparative analysis of torsional and bending stresses in two mathematical models of nickel–titanium rotary instruments: ProTaper versus ProFile. *J Endod* 2003: **29**: 15–19.
76. Seto BG, Nicholls JI, Harrington GW. Torsional properties of twisted and machined endodontic files. *J Endod* 1990: **16**: 355–360.
77. International Organization for Standardization. Dentistry—Root-canal Instruments—Part I: General requirements and test methods. ISO 3630-3631, 2008.
78. Council on Dental Materials, Instruments and Equipment. Revised American National Standards Institute/American Dental Association specification no. 28 for root canal files and reamers, type K (revised 1988).
79. 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 25/0.04 rotary instruments. *J Endod* 2008: **34**: 1406–1409.
80. 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**: 404–407.
81. Sattapan B, Nervo GJ, Palamara JE, Messer HH. Defects in rotary nickel–titanium files after clinical use. *J Endod* 2000: **26**: 161–165.
82. Cho OI, Versluis A, Cheung GS, Ha JH, Hur B, Kim HC. Cyclic fatigue resistance tests of nickel–titanium rotary files using simulated canal and weight loading conditions. *Restor Dent Endod* 2013: **38**: 31–35.
83. 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**: 1205–1209.
84. Montalvão D, Alçada FS. Numeric comparison of the static mechanical behavior between ProFile GT and ProFile GT series X rotary nickel–titanium files. *J Endod* 2011: **37**: 1158–1161.
85. Al-Hadlaq SM, Aljarbou FA, AlThumairy RI. Evaluation of cyclic flexural fatigue of M-wire nickel–titanium rotary instruments. *J Endod* 2010: **36**: 305–307.
86. Melton KN. NiTi based shape memory alloys. In: Duerig TW, Melton KN, Stöckel D, Wayman CM, eds. *Engineering Aspects of Shape Memory Alloys*. London: Butterworth-Heinemann, 1990: 21–25.
87. Bouska J, Justman B, Williamson A, DeLong C, Qian F. Resistance to cyclic fatigue failure of a new endodontic rotary file. *J Endod* 2012: **38**: 667–669.
88. Cheung GS, Darvell BW. Low-cycle fatigue of NiTi rotary instruments of various cross-sectional shapes. *Int Endod J* 2007: **40**: 626–632.
89. Cheung GS, Shen Y, Darvell BW. Effect of environment on low-cycle fatigue of a nickel–titanium instrument. *J Endod* 2007: **33**: 1433–1437.
90. Cheung GS, Darvell BW. Fatigue testing of a NiTi rotary instrument. Part 1: strain-life relationship. *Int Endod J* 2007: **40**: 612–618.
91. 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**: 1469–1476.
92. Plotino G, Grande NM, Cordaro M, Testarelli L, Gambarini G. Measurement of the trajectory of different NiTi rotary instruments in an artificial canal specifically designed for cyclic fatigue tests. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2009: **108**: 152–156.
93. Plotino G, Grande NM, Mazza C, Petrovic R, Testarelli L, Gambarini G. Influence of size and taper of artificial canals on the trajectory of NiTi rotary instruments in cyclic fatigue studies. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2010: **109**: 60–66.
94. Plotino G, Grande NM, Cordaro M, Testarelli L, Gambarini G. Influence of the shape of artificial canals on the fatigue resistance of NiTi rotary instruments. *Int Endod J* 2010: **43**: 69–75.
95. Pirani C, Cirulli PP, Chersoni S, Micele L, Ruggeri O, Prati C. Cyclic fatigue testing and metallographic analysis of nickel–titanium rotary instruments. *J Endod* 2011: **37**: 1013–1016.
96. Al-Sudani D, Grande NM, Plotino G, Pompa G, Di Carlo S, Testarelli L, Gambarini G. Cyclic fatigue of nickel–titanium rotary instruments in a double (S-shaped) simulated curvature. *J Endod* 2012: **38**: 987–989.
97. Gambarini G. Cyclic fatigue of nickel–titanium rotary instruments after clinical use with low- and high-torque endodontic motors. *J Endod* 2001: **27**: 772–774.
98. Galvão Barbosa FO, Ponciano Gomes JA, Pimenta de Araújo MC. Influence of previous angular deformation on flexural fatigue resistance of K3 nickel–titanium rotary instruments. *J Endod* 2007: **33**: 1477–1480.
99. Kim JY, Cheung GS, Park SH, Ko DC, Kim JW, Kim HC. Effect from cyclic fatigue of nickel–titanium rotary files on torsional resistance. *J Endod* 2012: **38**: 527–530.
100. Ullmann CJ, Peters OA. Effect of cyclic fatigue on static fracture loads in ProTaper nickel–titanium rotary instruments. *J Endod* 2005: **31**: 183–186.
101. Setzer FC, Böhme CP. Influence of combined cyclic fatigue and torsional stress on the fracture point of nickel–titanium rotary instruments. *J Endod* 2013: **39**: 133–137.