Influence of phase transformation on the torsional and bending properties of nickel–titanium rotary endodontic instruments

K. Miyai[1], A. Ebihara[1], Y. Hayashi[1], H. Doi[2], H. Suda[1] & T. Yoneyama[2,3]

[1]Pulp Biology and Endodontics, Department of Restorative Sciences, Graduate School; [2]Department of Metallurgy, Division of Biomaterials, Institute of Biomaterials and Bioengineering, Tokyo Medical and Dental University; and [3]Department of Materials Engineering, The University of Tokyo, Tokyo, Japan

Abstract

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. *International Endodontic Journal*, **39**, 119–126, 2006.

Aim To investigate the relationship between the functional properties and the phase transformation of nickel–titanium endodontic instruments.

Methodology Five types of rotary nickel–titanium endodontic instruments with a 0.30 mm diameter tip (EndoWave, HERO 642, K3, ProFile.06, and ProTaper) were selected to investigate torsional and bending properties, and phase transformation behaviour. A torsional test was performed according to ISO publication 3630-1, and maximum torque and angular deflection at fracture were measured. Bending load of the instruments was measured in a cantilever-bending test at 37 °C with the maximum deflection of 4.0 mm. A stainless steel K-file was used for reference. Phase transformation behaviour was measured by differential scanning calorimetry (DSC). From the DSC curve, transformation temperatures were calculated. Data were analysed by ANOVA and the Tukey–Kramer's test.

Results The maximum torsional torque values of HERO, K3 and ProTaper were significantly higher ($P < 0.05$) than those of EndoWave, ProFile and K-file. The K-files had the lowest torque value. Angular deflection at fracture was significantly higher ($P < 0.05$) for K-files than that for any nickel–titanium instrument. The bending load values of HERO and K3 were significantly higher ($P < 0.05$) than those of EndoWave, ProFile, ProTaper and K-file. The K-files had the lowest load value, although residual deflection remained. The transformation temperatures of HERO and K3 were significantly lower ($P < 0.05$) than those of EndoWave, ProFile and ProTaper.

Conclusions The functional properties of nickel–titanium endodontic instruments, especially their flexible bending load level, were closely related to the transformation behaviour of the alloys.

Keywords: bending property, differential scanning calorimetry, nickel–titanium alloy, phase transformation, rotary instruments, torsional property.

Received 10 September 2005; accepted 30 September 2005

Introduction

Nickel–titanium alloy is widely used in dentistry because of its superior mechanical properties, high corrosion resistance (Speck & Fraker 1980) and good biocompatibility (Castleman *et al.* 1976). Super-elasticity, associated with stress-induced martensitic transformation, is a unique property of this alloy (Yoneyama *et al.* 1992, 1993). In endodontics, nickel–titanium instruments with super-elasticity facilitate instrumentation of curved canals and efficient root canal preparation (Mandel *et al.* 1999, Schäfer & Florek 2003, Schäfer & Vlassis 2004). Various types of instruments with different designs, cross-sectional shapes and

Correspondence: Dr Karin Miyai, Pulp Biology and Endodontics, Department of Restorative Sciences, Graduate School, Tokyo Medical and Dental University, 5-45 Yushima 1-chome, Bunkyo-ku, Tokyo 113-8549, Japan (Tel.: +81 3 5803 5494; fax: +81 3 5803 5494; e-mail: k.miyai.endo@tmd.ac.jp).

manufacturing processes have been developed. The cross-sectional shape is important because it directly determines torsional and bending properties (Camps *et al.* 1995, Turpin *et al.* 2000, Berutti *et al.* 2003).

As the phase transformation behaviour of nickel–titanium alloy is influenced by numerous factors, including changes in its composition, machining characteristics and differences in heat treatment (Thompson 2000), the mechanical properties of the resulting nickel–titanium endodontic instruments differ. Clearly, this factor could affect the clinical performance achieved with these instruments. Commercial nickel–titanium endodontic instruments consist mainly of, nickel and titanium, in an equiatomic ratio (Schäfer *et al.* 2003). A high density of defects in the alloy caused by work-hardening could disturb the phase transformation, and the surface state of instruments is an important factor in fracture initiation (Kuhn *et al.* 2001). The mechanical properties and various phase transformation temperatures of nickel–titanium alloy are dependent on thermo-mechanical processing. For example, thermal treatments at approximately 400 °C before machining are reported to be effective in reducing work-hardening of the alloy (Kuhn & Jordan 2002).

Although the mechanical properties of nickel–titanium endodontic instruments have been reported, methods to evaluate their functional properties have yet to be established. Furthermore, the relationship between thermal behaviour and mechanical properties of nickel–titanium endodontic instruments has not been investigated sufficiently.

The purpose of this study was to elucidate the relationship between the functional properties and phase transformation of nickel–titanium endodontic instruments.

Materials and methods

Five types of rotary nickel–titanium endodontic instruments, EndoWave (FKG Dentaire, La-Chaux-de-Fonds, Switzerland), HERO 642 (Micro-Mega, Besançon, France), K3 (SybronEndo, West Collins, CA, USA), ProFile 0.06 (Dentsply Maillefer, Ballaigues, Switzerland) and ProTaper (Dentsply Maillefer), were selected for investigation. All the instruments that were selected were size 30 with a 0.06 taper, except for ProTaper. As ProTaper has a variable taper, size F3 with a 0.30 mm tip size was chosen. In the torsional and cantilever-bending tests, a size 30 stainless steel K-file (Zipperer, Munich, Germany) was used for reference. Five specimens of each instrument type were subjected to the following three tests.

Torsional test

The torsional test was carried out according to ISO 3630-1 (International Organization for Standardization 1992). The diameter of the instrument 3.0 mm from the tip was measured with a dial gauge (Teclock Co., Ltd, Nagano, Japan) five times, and each specimen was subsequently placed on a torsional testing apparatus (Orientec Co., Ltd, Tokyo, Japan). The handle of each specimen was removed and a portion 3.0 mm from its tip was clamped. Then, torsional moment (maximum torque) and angular deflection at fracture were measured. The specimen was rotated in a clockwise direction as viewed from the shank end, and the test speed was set at 19.1 deg s^{-1}.

Bending test

A cantilever-bending test was conducted with the use of a newly designed bending test machine (Fig. 1). The pole of the testing apparatus was combined with a load cell to measure the load on the specimen. A steel clamp was mounted on a movable stage connected with a displacement transducer to measure the deflection. The distance between the clamp edge and the specimen tip was 9.5 mm, and the initial loading point was 3.0 mm from the tip. The specimens were loaded until the deflection reached 4.0 mm, and then unloaded. The load on the specimen was measured during the loading and unloading process. The deflecting speed was approximately 0.1 mm s^{-1}. The specimens and apparatus were kept at 37 °C.

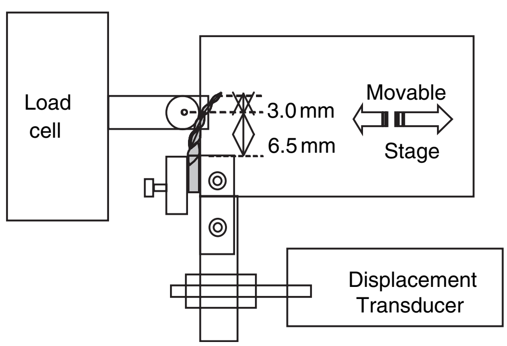

Figure 1 Schematic diagram of the cantilever-bending test device.

Differential scanning calorimetry (DSC) measurement

To investigate the phase transformation of nickel–titanium endodontic instruments, DSC was performed. Five specimens were subjected to DSC measurement. Each test specimen was taken from an instrument, which was cut into three to four segments, approximately 4 mm in length. The segments were weighed with an electronic balance and sealed in aluminum cells, which were placed in the measuring chamber of a differential scanning calorimeter (DSC-7000; ULVAC, Tokyo, Japan). The atmosphere of the chamber was argon gas, and alpha alumina powder was used as reference material. For each analysis, the specimen was initially heated from room temperature up to 100 °C, then cooled down to −100 °C in order to obtain the cooling curve, and subsequently heated again up to 100 °C to obtain the heating curve. The heating rate was 0.17 °C s^{-1}, and liquid nitrogen was used for the cooling process. From the DSC curve, the martensitic transformation-starting and -finishing (M_s, M_f) temperatures, reverse transformation-starting and -finishing (A_s, A_f) temperatures and the associated energy (Q) were calculated. The interpretation of the DSC diagram was based on the previous studies (Yoneyama *et al.* 1992, Bradley *et al.* 1996, Brantley *et al.* 2002a,b).

Data from the torsional and bending tests and DSC measurement were analysed using the one-way ANOVA for the detection of differences amongst the instruments. The Tukey–Kramer's test was performed as the *post hoc* test for detection of differences between the instruments. Statistical significance was set at $P < 0.05$.

Results

Torsional test

The cross-sectional width of each instrument type at the point 3.0 mm from the tips of the EndoWave, HERO, K3, ProFile, ProTaper and K-files was 0.490, 0.506, 0.490, 0.456, 0.580 and 0.370 mm, respectively. Figures 2 and 3 show the values of maximum torque and angular deflection at fracture of the specimens. The maximum torque values of HERO, K3 and ProTaper were significantly higher ($P < 0.05$) than those of EndoWave, ProFile and the K-file. The K-file had the lowest maximum torque. Angular deflection at fracture was significantly higher

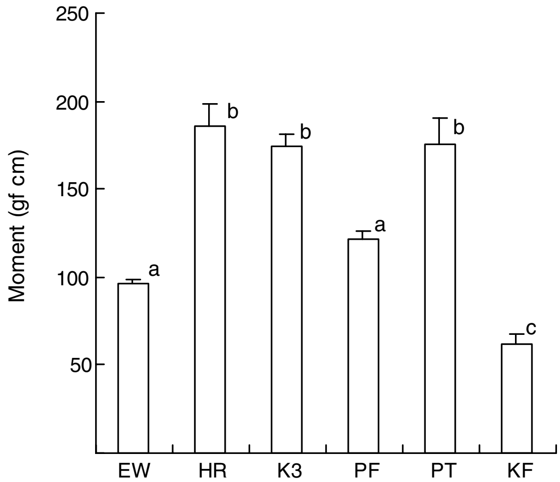

Figure 2 Maximum torque of each instrument in the torsional test. EW, EndoWave; HE, HERO; K3, K3; PF, ProFile; PT, ProTaper; KF, K-file. The bars with the same superscript letter were not significantly different ($P > 0.05$) $n = 5$.

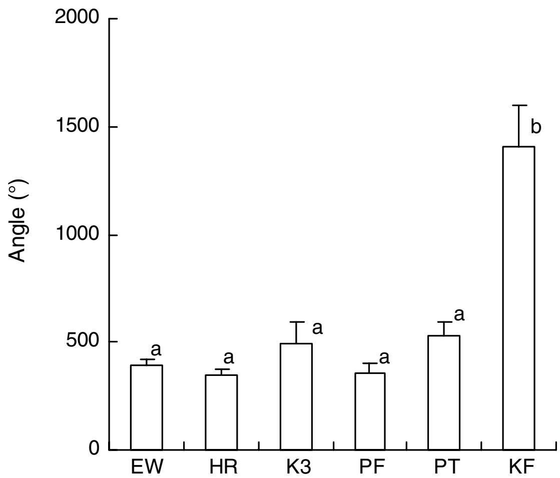

Figure 3 Angular deflection at fracture of each instrument in the torsional test. EW, EndoWave; HE, HERO; K3, K3; PF, ProFile; PT, ProTaper; KF, K-file. The bars with the same superscript letter were not significantly different ($P > 0.05$) $n = 5$.

($P < 0.05$) for the K-file than any of the nickel–titanium endodontic instruments.

Bending test

Figure 4 shows a schematic drawing of a typical load–deflection curve of a nickel–titanium endodontic instrument. The load deflection curve for each instrument

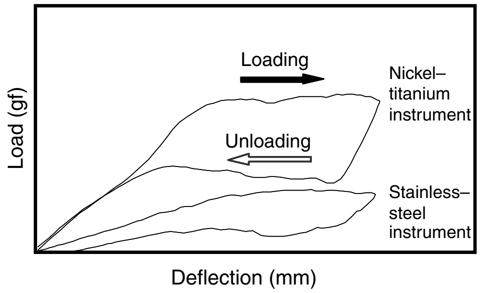

Figure 4 Typical load deflection curves of nickel–titanium and stainless steel instruments in the cantilever-bending test.

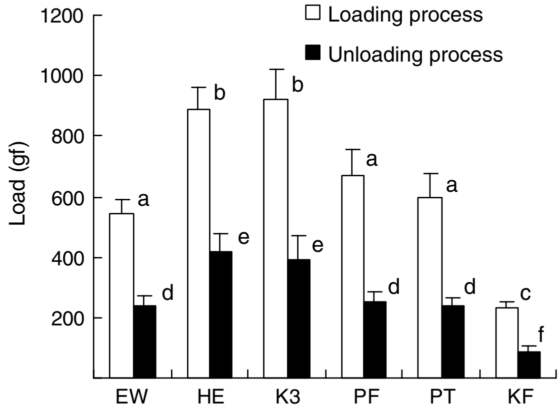

Figure 5 Bending load value at 3.0-mm deflection for each instrument. EW, EndoWave; HE, HERO; K3, K3; PF, ProFile; PT, ProTaper; KF, K-file. The bars with the same superscript letter were not significantly different ($P > 0.05$). $n = 5$.

showed a linear relationship up to 1.3–1.5 mm in the loading process. Above this level, the ratio gradually decreased, and the load level became almost constant above a deflection of 2.0 mm. In the unloading process, the load decreased rapidly when the deflection was reduced from 4.0 mm to approximately 3.5 mm. In the range of decreasing deflection from 3.5 to 1.3 mm, there was minimal change in the load. Permanent deformation values of nickel–titanium instruments did not exceed 0.01 mm, meaning that they recovered their original shape after being unloaded. On the other hand, the deflection of the K-file was elevated with the increasing load during the entire loading process. In the unloading process, the deflection decreased proportionally and the residual deflection was approximately 0.20 mm.

The bending load values at 3.0 mm deflection for HERO and K3 were significantly higher ($P < 0.05$) than those for EndoWave, ProFile, ProTaper and K-file, both in the loading and unloading processes. The K-file exhibited the lowest load value (Fig. 5).

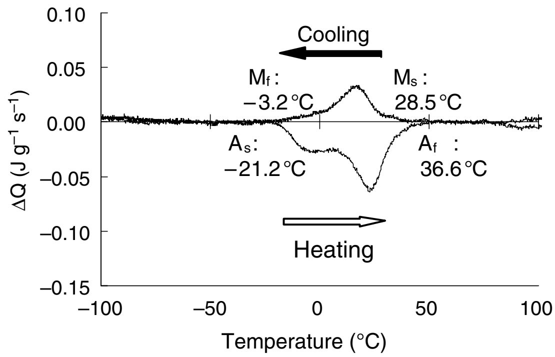

Figure 6 Typical differential scanning calorimetry diagram obtained from EndoWave.

DSC measurement

Figure 6 shows a typical DSC curve obtained from the EndoWave. In the DSC diagram, the exothermic reaction in the upper curve indicates the martensitic transformation in the cooling process, whilst the endothermic reaction in the lower curve is caused by the reverse transformation from martensitic phase to rhombohedral phase (R-phase) and/or austenitic phase in the heating process. There was a clear exothermic peak on the cooling curve of EndoWave. On the heating curve, there were two endothermic peaks; the smaller peak at approximately 0 °C corresponded to the initial transformation from martensitic phase to R-phase and the larger peak at approximately 25 °C corresponded to the transformation from R-phase to austenitic phase. In the DSC diagrams for ProFile and ProTaper, the transformation temperatures were almost the same as those for EndoWave, although the endothermic peaks at 0 °C were smaller. A typical DSC curve obtained from HERO is shown in Fig. 7. The cooling curve contained a broad, low exothermic peak at approximately −20 °C corresponding to the martensitic transformation. Two endothermic peaks at approximately −25 °C and −10 °C on the heating curve corresponded to the transformation from martensitic phase to R-phase and that from R-phase to austenitic phase. The transformation temperature of K3 was similar to that of HERO.

Table 1 summarizes the transformation temperatures and associated energy for each nickel–titanium endodontic instrument. These transformation temperatures were determined at the intersection of the

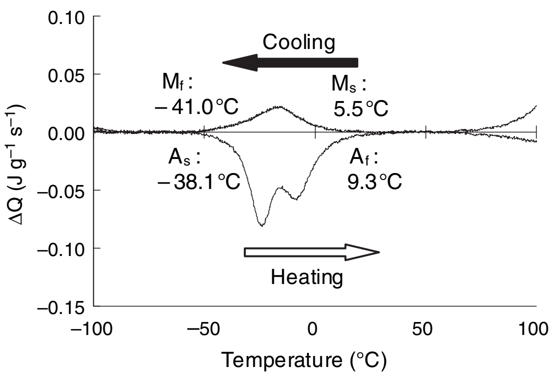

Figure 7 Typical differential scanning calorimetry diagram obtained from HERO.

baseline and the tangent of the peak slope. A_f temperatures of the instruments were between 5.4 and 36.6 °C, which were below the body temperature of 37 °C. Transformation temperatures of HERO and K3 were significantly lower ($P < 0.05$) than those of EndoWave, ProFile and ProTaper.

Discussion

Torsional property

Although the tip size of all the instruments used in this study was 0.30 mm, there were differences in the cross-sectional areas of the instruments 3.0 mm from the tip. It is difficult to precisely compare the mechanical properties of different types of nickel–titanium instruments, as they have different tapers and designs with a wide variety of cross-sectional shapes. However, in the current study, instruments with the same tip size were used.

The torsional torque and angular deflection at fracture reflect fracture resistance and ductility. Distortion and fracture of instruments occur in two ways: through torsional or flexural fatigue (Pruett *et al.* 1997, Sattapan *et al.* 2000). Torsional fracture can occur as a result of plastic deformation caused by a force exceeding the elastic limit of the metal. This occurs if a handpiece continues rotating whilst the tip of the instrument is restrained by the canal wall during instrumentation, especially in a narrow or curved canal (Hilt *et al.* 2000). Therefore, an instrument that has a higher value of maximum torque could be more resistant to torsional fracture.

Results of previous studies demonstrated a high correlation between the torque at fracture and instrument diameter (Camps & Pertot 1994, 1995, Marsicovetere *et al.* 1996, Ullmann & Peters 2005). Yared *et al.* (2003) reported that the torque at fracture increased significantly with increasing diameter, and that used instruments had lower torque at fracture compared with new ones. The cross-sectional shape is also an influential factor of torsional torque. Turpin *et al.* (2000) reported that the cross-sectional area of a triple helix (e.g. HERO) was around 30% larger than that of the triple-U (ProFile) and that a triple helix model had more than double the torsional inertia of a triple-U. Berutti *et al.* (2003) suggested that a difference in cross-sectional shape, convexity (ProTaper) or concavity (ProFile), affected the fracture pattern of instruments as well as the resistance to torsional fracture. In the current study, no clear correlation between the file diameter and the maximum torque was observed. The maximum torque of EndoWave was lower than that of HERO and K3, which had similar cross-sectional sizes. Despite a larger cross-sectional size, ProTaper had almost the same torque value as HERO and K3.

Bending property

Super-elasticity is one of the special characteristics of nickel–titanium alloy. When stress is applied to the alloy, it exhibits a unique behaviour compared with traditional alloys such as stainless steel. The phase of the alloy changes by stress-induced martensitic transformation at a temperature above the transformation temperature range. Owing to the twin deformation of

Table 1 Transformation temperatures and associated energy for Ni–Ti rotary instrument (mean ± SD, $n = 5$ for all instruments)

	Cooling process			Heating process		
	M_s (°C)	M_f (°C)	Q (J g^{-1})	A_s (°C)	A_f (°C)	Q (J g^{-1})
EndoWave	28.5 ± 1.2	−3.2 ± 2.0	1.4 ± 0.3	−21.2 ± 7.4	36.6 ± 1.7	1.3 ± 0.7
HERO 642	5.5 ± 2.6	−41.0 ± 1.9	1.2 ± 0.4	−38.1 ± 6.9	9.3 ± 1.5	1.1 ± 0.4
K3	9.2 ± 9.3	−43.7 ± 2.3	0.8 ± 0.2	−41.0 ± 3.3	5.4 ± 6.8	3.4 ± 2.1
ProFile.06	22.5 ± 1.9	−14.6 ± 6.1	1.4 ± 0.7	−20. 5 ± 3.0	29.6 ± 2.4	0.9 ± 0.3
ProTaper	19.1 ± 1.6	−10.4 ± 1.9	1.0 ± 0.5	−17.0 ± 8.1	32.1 ± 5.9	1.0 ± 0.2

this martensitic phase, nickel–titanium instruments maintain low stress levels even when a large deformation is applied and show little permanent deformation after the stress is removed. Such super-elasticity enables nickel–titanium endodontic instruments to exhibit excellent flexibility even in severely curved canals. Superior flexibility may reduce the risk of canal transportation during the preparation of curved canals (Walia *et al.* 1988, Thompson & Dummer 2000a,b, Schäfer & Florek 2003). Furthermore, the load on the cutting edges in curved canals is reduced, which in turn reduces the stress on the instrument and thus the possibility of fracture (Camps *et al.* 1995).

Schäfer *et al.* (2003) showed that cross-sectional configuration is the main factor affecting the bending properties of instruments. It is reasonable to hypothesize that the greater the diameter, cross-sectional area or taper of the instruments, the higher the bending stiffness of the instrument. Nevertheless, the present results demonstrated that the bending load values did not always depend only on the cross-sectional size. The bending load value of EndoWave instruments was lower than that of HERO and K3, although they have almost the same cross-sectional size. ProTaper had a low bending load value despite its large cross-sectional size. It has been suggested that other factors, such as the transformation characteristics of the alloy, would influence the bending property (Yoneyama *et al.* 1993) as well as the torsional property of the instruments.

Comparison between nickel–titanium and stainless steel instruments

There are many differences between nickel–titanium and stainless steel endodontic instruments. The taper of stainless steel instruments is standardized as 0.02 in ISO 3630-1. However, no standards are currently available for nickel–titanium rotary instruments, and instruments with various tapers have been developed. Nickel–titanium instruments with a taper greater than 0.02 can be used clinically owing to their super-elasticity. Instruments with greater taper enable the operator to shape root canals efficiently in the crown down technique (Bryant *et al.* 1999).

Beyond size 30, stainless steel instruments are likely to cause canal transportation owing to their high elastic modulus (Esposito & Cunningham 1995). In the cantilever-bending test of this study, K-files made of stainless steel had the lowest load values both in the loading and the unloading processes because of its small taper, whilst it remained permanently deformed after unloading as the recoverable range of K-files was less than those of the nickel–titanium instruments. The shape recovery of nickel–titanium instruments was considerably higher than that of stainless steel instruments because of their super-elasticity, and the bending stiffness of the former was higher than that of the latter because of the large taper.

Despite the many advantages of nickel–titanium endodontic instruments, unexpected fractures could occur during the use. The angular deflection at fracture is lower for nickel–titanium instruments than for stainless steel instruments, which possess excellent ductility. Instrument fractures can occur when the local torsional stress of the instruments is high even if the total angle of rotation is small. According to the results obtained from the torsional test, nickel–titanium instruments might fracture at approximately one rotation when the instrument tip is completely restrained. Furthermore, nickel–titanium instruments showed no visible deformation or signs of damage, so the operator may not be aware of plastic deformation, unlike stainless steel instruments (Mandel *et al.* 1999). Al-Fouzan (2003) found that small instruments experienced a higher percentage of distortion than large instruments.

Super-elasticity and phase transformation

At a temperature higher than the transformation temperature range, the crystal structure of nickel–titanium alloy is in austenitic phase of a CsCl type structure, whilst the structure is in martensitic phase of the monoclinic system at a lower temperature. When an external force is applied, stress-induced martensitic transformation from the austenitic phase to the martensitic phase occurs, and reverse transformation from the martensitic phase to the austenitic phase occurs in the unloading process. Thus, the repeated loading and unloading applied to nickel–titanium instruments during instrumentation cause repetitive phase transformation between the austenitic and martensitic phases.

The mechanical properties of nickel–titanium alloy associated with phase transformation are influenced by its composition, machining degree, thermal history, etc. Schäfer *et al.* (2003) found that nickel–titanium instruments are composed of an equiatomic ratio of nickel and titanium. However, the energy-dispersive spectroscopy applied may not detect slight changes in composition of the alloy that can result in a large difference in mechanical properties. It was also suggested that cold work and heat treatment should be controlled when processing nickel–titanium instruments (Yoneyama *et al.* 1992, Kuhn *et al.* 2001).

Differential scanning calorimetry, which is a general method to investigate the phase transformation behaviour of nickel–titanium instruments, was used in this study. In this analytical technique, the difference in thermal power supplied to a test specimen and an inert control material heated at the same rate is measured precisely (Bradley *et al.* 1996). The results of phase transformation temperatures (Table 1) indicated that nickel–titanium instruments were in austenitic phase (HERO, K3) or in a combination of austenitic and martensitic phases (EndoWave, ProFile, ProTaper) over the range between room and body temperatures. Therefore, these instruments can exhibit super-elastic flexibility during the clinical application.

The difference in transformation temperatures of nickel–titanium instruments is thought to have a considerable influence on their bending property. In the current study, the bending load value of nickel–titanium instruments correlated closely with their phase transformation temperature. The high load values in the loading process for HERO and K3 were probably caused by their low M_s temperature, as they required more stress to induce martensitic transformation. On the other hand, one of the main reasons for low load values in the unloading process for EndoWave, ProFile and ProTaper appeared to be their high A_f temperatures as their reverse transformation occurs at a relatively low stress level.

Two endothermic peaks were observed on the heating curves for EndoWave, ProFile and ProTaper instruments in the DSC diagrams. It is known that the elastic modulus of the austenitic phase is higher than that of the martensitic phase. Kuhn & Jordan (2002) described that how the elastic modulus of the intermediate R-phase was even lower than that of the martensitic phase. This two-step transformation through an R-phase may be another reason for the low load values of these instruments.

Conclusions

Functional properties and thermal behaviour of five commercial nickel–titanium rotary endodontic instruments were investigated using a torsional test, a cantilever-bending test and DSC. The following conclusions were drawn:

1. The maximum torsional torque values of HERO, K3 and ProTaper instruments were higher than those of EndoWave, ProFile and K-files. The K-file had the lowest torque. Angular deflection at fracture was higher for the K-file than any of the nickel–titanium instruments.

2. The bending load values at 3.0 mm deflection for HERO and K3 instruments were higher than those for EndoWave, ProFile, ProTaper and K-files, both in the loading and the unloading processes. The K-file had the lowest load value, and the residual deflection was approximately 0.20 mm.

3. A_f temperatures of HERO and K3 instruments (5.4–9.3 °C) were lower than those of EndoWave, ProFile and ProTaper (29.6–36.6 °C). They were below the body temperature of 37 °C.

4. Instruments that have low transformation temperatures (HERO, K3) tended to show higher maximum torque and higher bending load value than the instruments with high transformation temperatures (EndoWave, ProFile and ProTaper). Therefore, functional properties of nickel–titanium endodontic instruments may be closely correlated with the transformation behaviour of the alloy.

Acknowledgements

The authors would like to acknowledge Professor T. Hanawa of Tokyo Medical and Dental University for his guidance and discussion for this research. We also greatly thank Mr T. Takase, MANI, INC., for his cooperation.

References

Al-Fouzan KS (2003) Incidence of rotary ProFile instrument facture and the potential for bypassing in vivo. *International Endodontic Journal* **36**, 864–7.

Berutti E, Chiandussi G, Gaviglio I, Ibba A (2003) Comparative analysis of torsional and bending stresses in two mathematical models of nickel–titanium rotary instruments: ProTaper versus ProFile. *Journal of Endodontics* **29**, 15–9.

Bradley TG, Brantley WA, Culbertson BM (1996) Differential scanning calorimetry (DSC) analyses of superelastic and nonsuperelastic nickel–titanium orthodontic wires. *American Journal of Orthodontics and Dentofacial Orthopedics* **109**, 589–97.

Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH (2002a) Differential scanning calorimetric studies of nickel titanium rotary endodontic instruments. *Journal of Endodontics* **28**, 567–72.

Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH (2002b) Differential scanning calorimetric studies of nickel–titanium rotary endodontic instruments after simulated clinical use. *Journal of Endodontics* **28**, 774–8.

Bryant ST, Dummer PMH, Pitoni C, Bourba M, Moghal S (1999) Shaping ability of .04 and .06 taper ProFile rotary nickel–titanium instruments in simulated root canals. *International Endodontic Journal* **32**, 155–64.

Camps JJ, Pertot WJ (1994) Torsional and stiffness properties of canal master U stainless steel and nitinol instruments. *Journal of Endodontics* **20**, 395–8.

Camps JJ, Pertot WJ (1995) Torsional and stiffness properties of nickel–titanium K files. *International Endodontic Journal* **28**, 239–43.

Camps JJ, Pertot WJ, Levallois B (1995) Relationship between file size and stiffness of nickel–titanium instruments. *Endodontics and Dental Traumatology* **11**, 270–3.

Castleman LS, Motzkin SM, Alicandri FP, Bonawit VL (1976) Biocompatibility of Nitinol alloy as an implant material. *Journal of Biomedical Materials Research* **10**, 695–731.

Esposito PT, Cunningham CJ (1995) A comparison of canal preparation with nickel–titanium and stainless steel instruments. *Journal of Endodontics* **21**, 173–6.

Hilt BR, Cunningham CJ, Shen C, Richards N (2000) Torsional properties of stainless-steel and nickel–titanium files after multiple autoclave sterilizations. *Journal of Endodontics* **26**, 76–80.

International Organization for Standardization (1992) *Dental Root-Canal Instruments– Part 1: Files, Reamers, Barbed Broaches, Rasps, Paste Carriers, Explorers and Cotton Broaches*. Switzerland: ISO.

Kuhn G, Jordan L (2002) Fatigue and mechanical properties of nickel–titanium endodontic instruments. *Journal of Endodontics* **28**, 716–20.

Kuhn G, Tavernier B, Jordan L (2001) Influence of structure on nickel–titanium endodontic instruments failure. *Journal of Endodontics* **27**, 516–20.

Mandel E, Adib-Yazdi M, Benhamou LM, Lachkar T, Mesgouez C, Sobel M (1999) Rotary Ni–Ti profile systems for preparing curved canals in resin blocks: influence of operator on instrument breakage. *International Endodontic Journal* **32**, 436–43.

Marsicovetere ES, Burgess JO, Clement DJ, del Rio CE (1996) Torsional testing of the lightspeed nickel–titanium instrument system. *Journal of Endodontics* **22**, 681–4.

Pruett JP, Clement DJ, Carnes DL (1997) Cyclic fatigue testing of nickel–titanium endodontic instruments. *Journal of Endodontics* **23**, 7785.

Sattapan B, Palamara JEA, Messer HH (2000) Torque during canal instrumentation using rotary nickel–titanium files. *Journal of Endodontics* **26**, 156–60.

Schäfer E, Florek H (2003) Efficiency of rotary nickel–titanium K3 instruments compared with stainless steel hand K-Flexofile. Part 1. Shaping ability in simulated curved canals. *International Endodontic Journal* **36**, 199–207.

Schäfer E, Vlassis M (2004) Comparative investigation of two rotary nickel–titanium instruments: ProTaper versus Race. Part 1. Shaping ability in simulated curved canals. *International Endodontic Journal* **37**, 229–38.

Schäfer E, Dzepina A, Danesh G (2003) Bending properties of rotary nickel–titanium instruments. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology and Endodontics* **96**, 757–63.

Speck KM, Fraker AC (1980) Anodic polarization behavior of Ti-Ni and Ti-6Al-4V in simulated physiological solutions. *Journal of Dental Research* **59**, 1590–5.

Thompson SA (2000) An overview of nickel–titanium alloys used in dentistry. *International Endodontic Journal* **33**, 297–310.

Thompson SA, Dummer PMH (2000a) Shaping ability of Hero 642 rotary nickel–titanium instruments in simulated root canals: Part1. *International Endodontic Journal* **33**, 248–54.

Thompson SA, Dummer PMH (2000b) Shaping ability of Hero 642 rotary nickel–titanium instruments in simulated root canals: Part2. *International Endodontic Journal* **33**, 255–61.

Turpin YL, Chagneu F, Vulcain JM (2000) Impact of two theoretical cross-sections on torsional and bending stresses of nickel–titanium root canal instrument models. *Journal of Endodontics* **26**, 414–7.

Ullmann CJ, Peters OA (2005) Effect of cyclic fatigue on static fracture loads in ProTaper nickel–titanium rotary instruments. *Journal of Endodontics* **31**, 183–6.

Walia H, Brantley WA, Gernstein H (1988) An initial investigation of the bending and torsional properties nitinol root canal files. *Journal of Endodontics* **14**, 346–51.

Yared GM, Kulkarni GK, Ghossayn F (2003) An *in vitro* study of the torsional properties of new and used K3 instruments. *International Endodontic Journal* **36**, 764–9.

Yoneyama T, Doi H, Hamanaka H, Okamoto Y, Mogi M, Miura F (1992) Super-elasticity and thermal behavior of Ni–Ti alloy orthodontic arch wires. *Dental Materials Journal* **11**, 1–10.

Yoneyama T, Doi H, Hamanaka H, Yamamoto M, Kuroda T (1993) Bending properties and transformation temperatures of heat treated Ni–Ti alloy wire for orthodontic appliances. *Journal of Biomedical Research* **27**, 399–402.

