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Phase transformation behaviour and bending properties of hybrid nickel–titanium rotary endodontic instruments

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

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

Abstract

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Aim To investigate the bending properties of hybrid rotary nickel–titanium endodontic instruments in relation to their transformation behaviour.

Methodology Four types of nickel–titanium rotary endodontic instruments with different cross-sectional shapes (triangular-based and rectangular-based) and different heat treatment conditions (super-elastic type and hybrid type with shape memory effect) were selected to investigate bending properties and phase transformation behaviour. Bending load of the instruments was measured in a cantilever-bending test at 37 °C with the maximum deflection of 3.0 mm. A commercial rotary instrument, ProFile (PF; Dentsply Maillefer, Ballaigues, Switzerland) was used as a reference for the bending test. Phase transformation temperatures were calculated from the diagrams obtained from differential scanning calorimetry. Data were analysed by ANOVA and Scheffe's test.

Results The bending load values of the hybrid type that had undergone additional heat treatment at the tip were significantly lower ($P < 0.05$) than those of the super-elastic type with no additional heat treatment. The bending load values of rectangular-based cross-sectional shaped instruments were significantly lower ($P < 0.05$) than those of triangular-based cross-sectional shaped instruments. Phase transformation temperatures (M_s and A_f points) of the hybrid type were significantly higher ($P < 0.05$) than the super-elastic type. The M_f and A_s points of the tip part were significantly higher ($P < 0.05$) than those of the whole part of the hybrid instrument.

Conclusions Additional heat treatment of hybrid nickel–titanium instruments may be effective in increasing the flexibility of nickel–titanium rotary instruments.

Keywords: bending property, heat treatment, hybrid instruments, Ni–Ti alloy, phase transformation.

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Introduction

One of the major reasons for using nickel–titanium (Ni–Ti) alloy for root canal instruments is that it exhibits greater flexibility compared with other alloys such as stainless steel (Walia *et al.* 1988). The flexibility of Ni–Ti alloy offers distinct clinical advantages for the preparation of curved root canals (Walia *et al.* 1988, Glosson *et al.* 1995, Pruett *et al.* 1997, Brantley *et al.* 2002a,b). Many manufacturers have developed and

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Correspondence: Dr Yohsuke Hayashi, 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: y.hayashi.endo@tmd.ac.jp).

designed new Ni–Ti rotary endodontic instruments that have different features, such as various cross-sectional shapes, pitches and tapers, and these instruments have been applied in clinical settings. Previously, there have been many studies to evaluate the shaping ability of nickel–titanium rotary endodontic instruments (Bryant *et al.* 1998, Mandel *et al.* 1999, Peters *et al.* 2003, da Silva *et al.* 2005).

Nickel–titanium alloy has special characteristics of super-elasticity and shape memory. Super-elasticity and the shape memory effect are derived from the martensitic and reverse transformations with super-elasticity being observed at temperatures above the reverse transformation finishing temperature termed the A_f point (Yoneyama *et al.* 1993). The mechanical properties of Ni–Ti alloy are easily influenced by small changes in composition, impurities and heat treatment conditions (Bahia *et al.* 2005). In particular, super-elasticity and shape memory are strongly affected by heat treatment (Yoneyama *et al.* 1992, 1993, 1999, Thompson 2000, Kuhn *et al.* 2001, Kuhn & Jordan 2002). It has also been reported that the technique of partial heat treatment is relatively difficult for Ni–Ti alloys (Yoneyama *et al.* 1993, 2002, Thompson 2000). However, in a previous study, Yoneyama *et al.* (1993) reported that heat-treated Ni–Ti alloy wires had greater flexibility than that of the original alloy wire.

Not only the mechanical properties of Ni–Ti alloy, but also the configuration of the instruments, especially the cross-sectional shape, are important factors for the performance of rotary endodontic instruments (Schäfer & Florek 2003, Schäfer & Lohmann 2002, Berutti *et al.* 2003). Instruments are available in a variety of cross-sectional shapes including triangular such as ProTaper (Dentsply Maillefer, Ballaigues, Switzerland) and RaCe (FKG Dentaire, La Chauxde-Fonds, Switzerland), and modified triangular such as K3 (Sybron Endo, West Collins, CA, USA). A modified rectangular cross-sectional shape design has also been developed for stainless-steel hand instruments, RT files (MANI Inc., Utsunomiya, Japan).

Recently, a rotary endodontic instrument with hybrid technologies, which combines super-elasticity and shape memory, has been developed for clinical use as the NRT file (MANI Inc.). This file has the same cross-sectional shape design as the RT file. However, the difference in mechanical properties between this hybrid Ni–Ti alloy rotary instrument and traditional ones has not yet been investigated. The aim of this study was to investigate the bending properties of hybrid rotary endodontic instruments in relation to their transformation behaviour.

Materials and methods

Specimens

Two prototype Ni–Ti rotary instruments that had undergone different treatments were used; super-elastic type (type 1) and hybrid type with both shape memory and super-elasticity (type 2), as shown in Fig. 1. For the type 2 instrument, an additional heat treatment was performed at a length of 5 mm from the instrument tip before the final machining of the flutes. After the heat treatment, two different cross-sectional shapes; triangular-based (type T) and rectangular-based (type R), were given to types 1 and 2 instruments, as shown in Fig. 2. Consequently type 2R specimens had a configuration similar to commercial NRT files. In addition to these four types of rotary instrument (types 1T, 1R, 2T and 2R), a commercial Ni–Ti rotary instrument, ProFile (Fig. 2), was used as a reference for the bending test.

For the cantilever-bending test, size-30 instruments with 0.04 and 0.06 tapers were used. For the differential scanning calorimetry (DSC) measurement, size-30 instruments with 0.06 taper were used. Five specimens for each instrument type were subjected to the following two tests.

DSC measurement

To investigate the phase transformation of Ni–Ti endodontic instruments, DSC measurements were performed using a DSC-7000 differential scanning

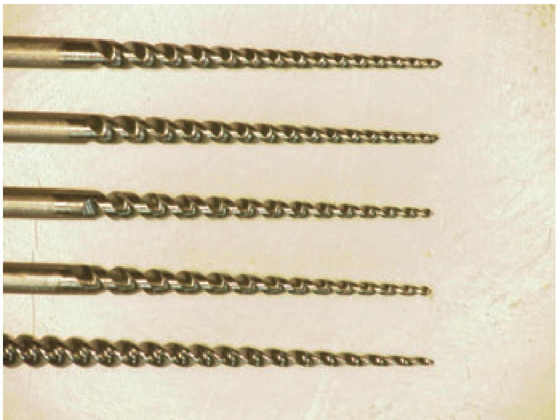

Figure 1 Experimental files (upper to lower: 1T, 2T, 1R and 2R) and ProFile (bottom). Original magnification: 20×.

Figure 2 Cross-sectional shapes of instruments at 3 mm from the tip (left: T, centre: R, right: PF).

calorimeter (ULVAC, Tokyo, Japan). Each instrument was cut into segments and enclosed in aluminium cells. The entire blade section of four types of specimen, 1T, 1R, 2T and 2R, was used for the DSC measurement. In addition, the 5-mm tip of 2R instruments was tested as Tip specimens.

Measurements were made in the temperature range from +100 to −100 °C, under an argon gas atmosphere. Liquid nitrogen was used for the cooling process. During the heating process, the rate of temperature rise was controlled at 0.17 °C s^{-1}. Twenty milligrams of alpha alumina powder was also enclosed in an aluminium cell and used as the reference material. The following transformation temperatures were calculated from the DSC curves obtained for each of the specimens: martensitic transformation starting point (M_s), martensitic transformation finishing point (M_f), reverse transformation starting point (A_s), and reverse transformation finishing point (A_f). The interpretation of the DSC diagram was based on previous studies (Yoneyama *et al.* 1992, Bradley *et al.* 1996, Brantley *et al.* 2002a,b, Hayashi *et al.* 2005, Miyai *et al.* 2006).

Cantilever-bending test

In this study, a cantilever-bending test device (Fig. 3) was used at 37 °C. Each instrument was held at a point 7.0 mm from the tip, and the loading point was 0.5 mm from the tip. The maximum deflection was set at 3.0 mm. The loading and unloading speed was approximately 0.1 mm s^{-1}. The bending load value at 2.4 mm deformation in the loading process was used as the parameter for evaluation. The bending load was analysed by one-way analysis of variance and Scheffe's *post hoc* test. The statistical significance was set at a confidence level of 95%.

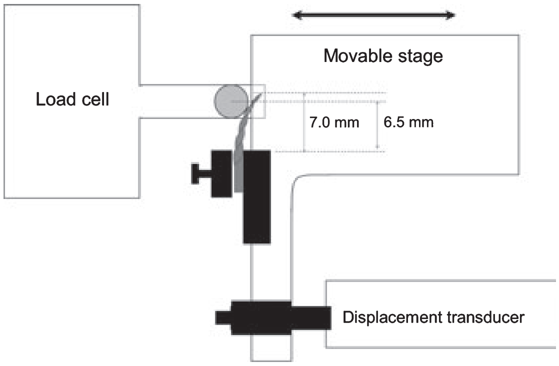

Figure 3 Schematic drawing of the original cantilever-bending test device viewed from above.

Results

Transformation temperatures

Figure 4a–c show typical DSC traces for the 1R, 2R, and Tip specimens, respectively. For each DSC trace, the upper and lower lines indicate thermal changes in the cooling and heating process, respectively. An exothermic peak was observed for each specimen during the cooling process, which was located at a higher temperature for the Tip than for type 1 specimens. The type 2 specimens consisted of two exothermic peaks, which were located at temperatures similar to those for the type 1 and Tip specimens. Endothermic reactions during the heating process resulted in two peaks on every DSC trace, and the peak at higher temperature was relatively larger for the Tip specimens than for the other specimens.

Table 1 shows the transformation temperatures obtained from the DSC traces for each specimen. M_s and A_f points for the type 2 and Tip were higher than those for the type 1, whilst M_f and A_s points for the Tip were higher than those for the types 1 and 2. There were no statistically significant differences in the transformation temperatures between the specimens with different cross-sectional shapes.

Bending property

Figure 5 shows a typical load–deflection curve of a 0.06 tapered Ni–Ti rotary instrument obtained from the cantilever-bending test. Figure 6 shows the bend-

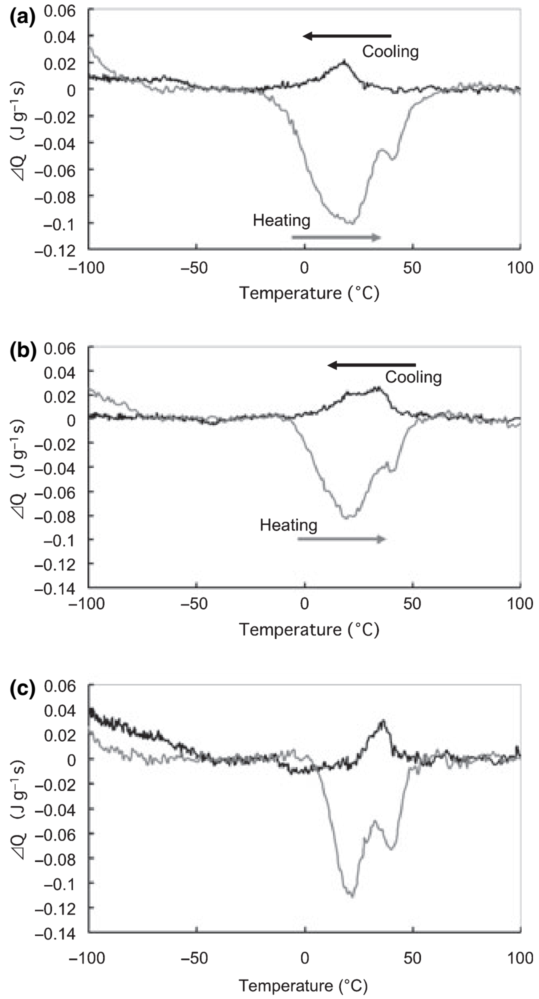

Figure 4 (a) Typical differential scanning calorimetry diagram obtained from super-elasticity instrument (1R). (b) Typical differential scanning calorimetry diagram obtained from shape memory effect instrument (2R). (c) Typical differential scanning calorimetry diagram obtained from Tip group.

Table 1 Phase transformation temperatures for Ni–Ti rotary instruments (mean ± SD). M_s: martensitic transformation starting point, M_f: martensitic transformation finishing point, A_s: reverse transformation starring point, A_f: reverse transformation finishing point. Different superscript letters in each column represent no statistically significant difference at $P > 0.05$ ($n = 5$ for all instruments)

	M_s (°C)	M_f (°C)	A_s (°C)	A_f (°C)
1T	$+30.6\pm2.5^a$	-3.5 ± 1.6^a	-10.5 ± 1.0^a	$+39.2\pm2.2^a$
1R	$+28.8\pm0.6^a$	$-2.0\pm1.3^{a,b}$	-9.3 ± 0.8^a	$+38.6\pm3.8^a$
2T	$+40.9\pm4.8^b$	$+4.6\pm3.1^{b,c}$	-10.8 ± 1.0^a	$+49.2\pm4.6^b$
2R	$+43.8\pm1.3^b$	$+6.6\pm5.8^c$	-7.1 ± 3.0^a	$+50.4\pm1.5^b$
Tip	$+42.8\pm1.3^b$	$+22.5\pm3.7$	$+4.3\pm4.8$	$+49.9\pm1.6^b$

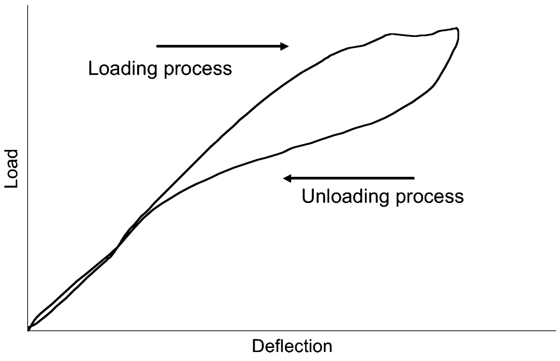

Figure 5 Typical load–deflection curve of nickel–titanium instrument (2R) in the cantilever-bending test.

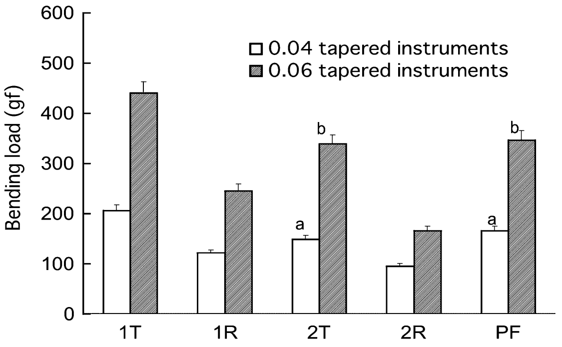

Figure 6 Bending loads of 0.04 and 0.06 tapered instruments. The same superscript letter represents no statistically significant difference ($P > 0.05$, $n = 5$).

ing load values for 0.04 tapered specimens at 2.4 mm displacement during the loading process. Those instruments with a triangular-based cross-sectional shape displayed a higher bending load than those that were rectangular-based, for both heat treatment conditions (types 1 and 2). With respect to the heat treatment conditions, type 1 showed a higher bending load than type 2 for both cross-sectional shapes. Significant differences were recognized amongst all groups ($P < 0.05$) except between 2T and PF.

Figure 7 shows the bending load for 0.06 tapered instruments at 2.4 mm displacement. For this taper, type 1 and type T displayed a higher bending load than type 2 and type R, respectively, which was similar to that observed for the 0.04 tapered specimens. The bending load values for 0.06 tapered instruments were approximately twice as much as those for the 0.04 tapered ones.

Discussion

Ni–Ti alloy has special mechanical properties of super-elasticity and a shape memory effect. Super-elasticity is derived from the stress-induced martensitic transformation caused by an externally applied force. The flexibility of Ni–Ti rotary endodontic instruments is based on this super-elasticity.

The transformation temperature is one of the most important factors that influences the mechanical properties of Ni–Ti alloy. There are several factors affecting the phase transformation of Ni–Ti alloy, such as composition, degree of machining and heat treatment. Miyai *et al.* (2006) found that there were differences amongst the transformation temperatures of commercial Ni–Ti rotary instruments, which caused differences in their torsional and bending properties.

DSC is one of the major methods to measure the transformation behaviour of Ni–Ti alloys. From the DSC results of this study, the M_s and A_f points of hybrid instruments (types 2T and 2R) were higher than those of the super-elastic instruments (types 1T and 1R), whilst there was no difference in the A_s points of these groups. The M_s and A_f points of the Tip were similar to those of types 2T and 2R, and therefore the difference between the transformation temperatures of the hybrid and super-elastic instruments was due to the heat-treatment of the Tip part. In addition, the M_f and A_s points of Tip were clearly higher than those of the whole part of the hybrid instruments. This indicates that the hybrid instruments consisted of parts with different transformation characteristics. As for the two thermal peaks observed in the heating process of the DSC traces, there may exist two possible reasons; appearance of an intermediate R-phase, and a peak shift caused by the additional heat treatment at the instrument tip.

The test methods for endodontic instruments are standardized according to ISO specification No.3630-1 (ISO 1992), and there are some reports on the mechanical properties of Ni–Ti instruments based on ISO 3630-1 (Peters & Barbakow 2002, Schäfer *et al.* 2003, Ullmann & Peters 2005). Those test methods were designed for stainless steel endodontic devices, and other test methods have been introduced to evaluate important characteristics of Ni–Ti rotary instruments, such as fatigue behaviour and shaping ability. In this study, a cantilever-bending test was performed at body temperature, and the loading point was set at 0.5 mm from the tip to investigate the bending property of the tip part of the instrument. The observed bending properties of the hybrid and super-elastic instruments revealed that the bending load of the hybrid instruments was lower than that of the super-elastic instruments for each taper size and cross-sectional shape. The main reason for this difference may be because of the difference in the M_s point, i.e. low load values for the hybrid instruments are caused by a higher M_s point at the tip part, because less stress is required to induce martensitic transformation.

The configuration of rotary instruments is also an important factor to determine their mechanical properties. Two different cross-sectional shapes were used for the Ni–Ti rotary instruments in this study; the triangular-based shape, similar to the traditional ProFile instrument, and the rectangular-based, which was reported to show better shaping ability in a previous study (Etori *et al.* 2002). The bending load values of the instruments with a rectangular cross-sectional shape were lower than those with a triangular cross-sectional shape. There was no significant difference between the bending load values of the hybrid instruments with a triangular cross-sectional shape and the ProFile instrument. However, the additional heat treatment for the hybrid instrument was effective for reducing the bending load of the instruments with a rectangular cross-sectional shape, which exhibited superior bending properties that would allow the use of severely curved root canals.

Conclusions

The phase transformation behaviour and bending properties of hybrid rotary endodontic Ni–Ti instruments were investigated using DSC and a cantilever-bending test. The following conclusions were drawn:

1. The bending load values of hybrid Ni–Ti instruments that had undergone additional heat treatment at the tip were lower than those of original Ni–Ti instruments with no additional heat treatment.

2. The bending load values of Ni–Ti instruments with rectangular-based cross-sectional shapes were lower than those of Ni–Ti instruments with triangular-based cross-sectional shapes, including the ProFile instrument.

3. The M_s and A_f points of the hybrid instruments were higher than those of original Ni–Ti instruments with no additional heat treatment.

4. The M_f and A_s points of the Tip part were higher than those of the whole part of the hybrid instrument, which indicated that the hybrid instrument consisted of parts with different transformation characteristics.

5. The additional heat treatment of the hybrid Ni–Ti instruments may be effective to increase the flexibility of Ni–Ti rotary instruments.

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