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Phase transformation behaviour and bending property of twisted nickel–titanium endodontic instruments

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Abstract

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Aim To investigate the relationship between phase transformation behaviour and bending property of nickel–titanium endodontic instruments manufactured by a twisting process.

Methodology The phase transformation behaviour and bending property of Twisted Files (TF; SybronEndo, Orange, CA, USA) and K3 (SybronEndo) with.06 taper and size 30 tip were investigated. K3 was used as control group. Phase transformation behaviour was estimated by differential scanning calorimetry (DSC). Transformation temperatures were calculated from the DSC curve. Bending load of the instruments was measured by cantilever-bending test at 37 °C. Data were analysed by Student's *t*-test and Mann–Whitney *U*-test.

Results The phase transformation temperatures of TF were significantly higher ($P < 0.05$) than those of K3. The bending load values were significantly lower for TF than that of K3 ($P < 0.05$), both in the elastic and super-elastic ranges.

Conclusions The new method of manufacturing NiTi instruments by twisting coupled with heat treatment might contribute to the increased phase transformation temperatures and superior flexibility.

Keywords: bending property, differential scanning calorimetry, manufacturing process, nickel–titanium alloy, Twisted File.

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Introduction

Nickel–titanium (NiTi) instruments were first applied in the field of endodontics in 1988 (Walia *et al.* 1988). Since then, they have played an increasingly important role in endodontics. It is known that the super-elasticity and shape memory effect of these instruments are derived from martensitic transformation. At a temperature above the transformation temperature range, NiTi alloy is composed of austenite whilst at a lower temperature it consists of martensite (Huang *et al.* 2003). A decrease in temperature can trigger phase transformation from austenite to martensite (termed martensitic transformation), and vice versa (reverse transformation). This phenomenon could also be triggered by stress (Daly *et al.* 2007). When temperature is higher than the transformation temperature and the NiTi alloy is composed of austenite, loading and unloading can cause forward martensitic transformation (i.e. stress-induced martensitic transformation) and reverse transformation (Miyazaki & Otsuka 1989, Thompson 2000). Furthermore, the rhombohedral (R-) phase transformation, also thermo-elastic, precedes the

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martensitic transformation under certain conditions (Miyazaki & Otsuka 1986). This reversible thermoelastic martensitic transformation is the main reason for increased flexibility of NiTi instruments over traditional stainless steel ones, which facilitates instrumentation of curved root canals (Walia *et al.* 1988, Kazemi *et al.* 2000, Perez *et al.* 2005).

Phase transformation behaviour has an impact on the mechanical properties of NiTi instruments (Yoneyama *et al.* 1992, 2002, Miyai *et al.* 2006, Hayashi *et al.* 2007, Yahata *et al.* 2009) and the former is easily influenced by factors including chemical composition, heat treatment and manufacturing processes (Miyazaki *et al.* 1982, Thompson 2000).

Recently, a new manufacturing process was developed by SybronEndo (Orange, CA, USA) to create a NiTi endodontic instrument named the Twisted File (TF). According to the manufacturer, TF instruments are developed by transforming a raw NiTi wire in the austenite to R-phase through a thermal process. In the R-phase, the NiTi blank is twisted along with repeated heat treatment, and after additional thermal procedures to maintain its new shape, the instrument is converted back to austenite, which is super-elastic once stressed. The manufacturer claims that this proprietary twisting process with concurrent heat treatment and protection of the crystalline structure imparts superior flexibility and resistance to fatigue. TF instruments are significantly more resistant to cyclic fatigue than ground ones (Gambarini *et al.* 2008a,b, Kim *et al.* 2010, Oh *et al.* 2010).

However, the lack of knowledge on the phase transformation behaviour of TF might impede understanding of the influence of the new manufacturing process on the mechanical properties. The aim of the present study was therefore to investigate the thermal behaviour of TF in relation to its bending property. The null hypothesis was that there would be no difference between the two types of instruments with regards to their thermal behaviour and bending property.

Materials and methods

Differential scanning calorimetry

TF and K3, both manufactured by SybronEndo, were evaluated. The blade sections of TF and K3 instruments, with constant 0.06 taper and size 30 tip, were cut into 2- to 3-mm-long specimens weighing 20 mg each. K3 was selected as the control group. The specimens were sealed in aluminium cells, which were placed in the measuring chamber of a differential scanning calorimeter (DSC-60; Shimadzu, Kyoto, Japan). With an empty aluminium cell placed as the reference, the chamber was filled with argon gas. Liquid nitrogen acted as coolant and the heating and cooling rate was set to 10 °C min^{-1}. The temperature was increased from room temperature to 100 °C, and then reduced to −100 °C to obtain a cooling curve. The subsequent increase back to 100 °C enabled heating curve recording. Five specimens of each group were tested.

The interpretation of the DSC diagram was based on previous studies (Miyai *et al.* 2006, Hayashi *et al.* 2007, Yahata *et al.* 2009), in which the transformation temperatures were obtained from the intersection between extrapolation of the baseline and maximum gradient line of the lambda-type DSC curve. The martensitic transformation-starting and -finishing points (M_s, M_f) and reverse transformation-starting and -finishing points (A_s, A_f) were determined. Furthermore, individual or combined peak areas (enthalpies) were also calculated from the DSC plots.

Bending test

Another 20 size 30, .06 taper TF and K3 instruments ($n = 10$) were tested using a cantilever-bending test model. The experimental setting was based on previous studies (Miyai *et al.* 2006, Yahata *et al.* 2009) and a universal testing machine (Autograph AG-IS; Shimadzu) was used to apply and record the load. After cutting off the handle, each specimen was clamped at 9.5 mm from the tip, and the loading point was set at 3.0 mm from the tip. The loading speed was 1.0 mm min^{-1}, and once the deflection reached 3.0 mm, the unloading process was started. The bending loads at deflections of 0.5 mm and 2.0 mm in the loading process were recorded. The temperature of the specimens and apparatus was maintained at 37 °C.

Data analysis

The data from the DSC test had normal distributions and homogeneity of variances according to the Levene test and the results for the bending test did not. Student's *t*-test was used to compare DSC data of the two groups and Mann–Whitney *U*-test was used to detect the difference of the bending load values between TF and K3. The statistical significance was set at $P = 0.05$.

Results

DSC measurement

From the typical DSC curve of TF (Fig. 1), one clear exothermic peak was observed on the cooling curve. Two endothermic peaks were observed on the heating curve; the first peak corresponded to the initial transformation from martensite to R-phase and the second peak corresponded to the transformation from R-phase to austenite. However, the peaks overlapped, making it difficult to separate them. Thus, the phase transformation temperatures were determined from tangent lines where the DSC curve deviates from and returns to the baselines, denoted as A_s and A_f, respectively. As shown in Fig. 1, the typical DSC curve for K3 exhibited single and low peak on cooling and heating, respectively. This represents the martensitic and reverse transformation between austenite and martensite. Comparison between the two diagrams reveals that the thermal peaks were located at higher temperature for TF than K3, and the area under the peaks was larger for TF than for K3.

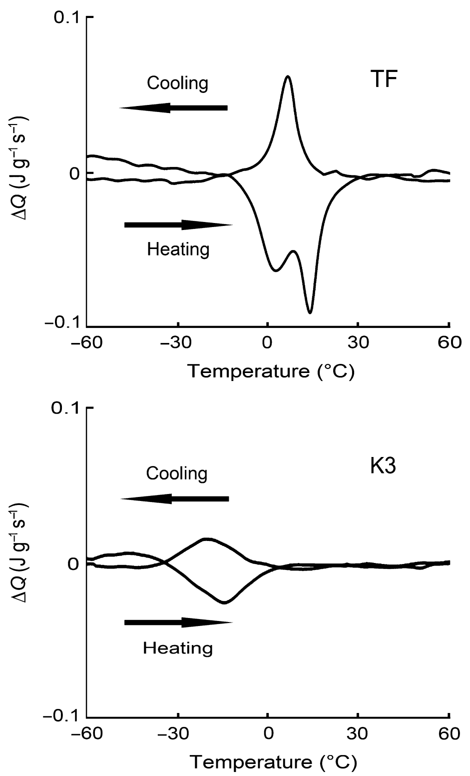

Figure 1 Typical differential scanning calorimetry (DSC) curves for Twisted Files (TF) and K3.

Table 1 summarizes the transformation temperatures and associated energy for the two instruments. The A_f and M_s temperatures for both instruments were lower than the body temperature of 37 °C. All the phase transformation points as well as ΔH values were significantly higher for TF than K3 ($P < 0.05$).

Bending test

Figure 2 illustrates a schematic drawing of typical load–deflection curves of TF and K3. The curve for TF showed a linear relationship initially, representing the elastic deformation. Then it extended into a plateau, representing the stress-induced phase transformation. In the following unloading process, the load reached a plateau swiftly and the latter indicated the reverse transformation. Subsequently, elastic unloading occurred, leaving a small residual deflection. The stress hysteresis (difference between the loading and unloading plateaus) of TF was small. The load–deflection curve of K3 was also composed of initial and final linear regions and two plateaus except that the plateaus were located much higher than those of TF and the stress hysteresis was much larger. The permanent deformation was also small for K3.

Table 2 shows bending loads at deflection of 0.5 and 2.0 mm, corresponding to the elastic range and superelastic range, respectively. TF revealed significantly lower load values than K3 in both of the ranges ($P < 0.05$).

Discussion

Data analysis suggested that the null hypothesis was rejected. Significant differences existed between TF and K3 with regards to their thermal behaviour and bending property.

Owing to the thermo-elastic nature of R-phase and martensitic transformation of NiTi alloy, thermal changes can be used to represent the differences in the free energies amongst austenite, R-phase and martensite. DSC, which provides a microcalorimetric determination of the reference material and specimen, indicates which of the three phases will be present at a given temperature. It is generally used to determine the transformation behaviour of NiTi alloy and the crystallographic structure of NiTi instruments (Bradley *et al.* 1996, Brantley *et al.* 2002a, American Society of Testing and Materials 2005).

From this study, M_s and A_f were lower than the body temperature of 37 °C for both TF and K3 specimens

Table 1 Phase transformation temperatures and associated energy of Twisted Files (TF) and K3 (mean ± SD, n = 5)

	Cooling process			Heating process		
	M_s (°C)	M_f (°C)	ΔH (J g^{-1})	A_s (°C)	A_f (°C)	ΔH (J g^{-1})
TF	9.37 ± 1.40[a]	−3.91 ± 1.86[a]	3.26 ± 0.32[a]	−8.22 ± 1.31[a]	18.88 ± 1.70[a]	8.71 ± 3.09[a]
K3	−2.02 ± 2.40	−30.86 ± 5.50	1.90 ± 0.33	−34.80 ± 5.90	3.88 ± 3.21	3.93 ± 0.42

[a]Statistically significant difference compared with K3 (P < 0.05).

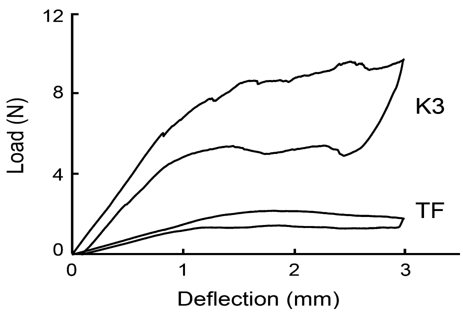

Figure 2 Typical load–deflection curves for Twisted Files (TF) and K3.

Table 2 Bending load values at deflections of 0.5 mm and 2.0 mm for Twisted Files (TF) and K3 (mean ± SD, n = 10)

	0.5-mm deflection (N)	2.0-mm deflection (N)
TF	0.84 ± 0.14[a]	2.33 ± 0.28[a]
K3	3.33 ± 0.39	7.76 ± 1.17

[a]Statistically significant difference compared with K3 (P < 0.05).

(Table 1), meaning that these instruments consist mainly of austenite in the oral environment. These instruments would exhibit super-elastic property during clinical application.

The transformation temperature is one of the most important factors that characterize the NiTi instrument. Heat treatment and work hardening during manufacturing are believed to greatly influence the thermal behaviour of NiTi instruments (Bradley *et al.* 1996, Brantley *et al.* 2002a, Kuhn & Jordan 2002, Hayashi *et al.*2007, Yahata *et al.*2009).

Conventionally, the raw NiTi alloy is subjected to vacuum casting, hot forging, rolling and drawing to create the wire. This wire is further drawn and repeatedly annealed to achieve the final diameter, which successively is subjected to grinding to obtain the configuration of the instrument (Thompson 2000). The NiTi instruments have to be machined rather than twisted because the super-elasticity of the alloy deprives it of its ability to maintain permanent deformation of the spiral configuration. Attempts to twist instruments in the conventional way would probably result in instrument fracture (Schäfer 1997). However, in the case of TF, maintaining the NiTi alloy in R-phase by heat treatment enables the twisting process. R-phase possesses lower shear modulus than martensite and austenite, and the transformation strain for R-phase transformation is less than one-tenth that of martensitic transformation (Wu *et al.* 1990, Otsuka & Wayman 1998). Thus, it is easier to apply plastic deformation to R-phase because lower stress would be required.

From the DSC results of this study, all the four tested phase transformation points were significantly higher for TF than those for K3 (P < 0.05) (Table 1). This is indicative of the variations in the processing of these instruments. Although the exact thermomechanical history remains unknown, it was suggested that K3 is manufactured by machining cold worked wires without thermal treatment (Zinelis *et al.* 2010). In this case, the manufacturing of NiTi blank and the subsequent machining procedure induce a large amount of cold work in K3, which might lower the phase transformation temperatures. In contrast, the twisting process avoids machining defects and it was well documented that heat treatment might modify the transformation temperature by releasing crystal lattice defects and diminishing internal strain energy (Miyazaki *et al.* 1982, Miyazaki & Otsuka 1986, Todoroki & Tamura 1987, Huang & Liu 2001, Yoneyama *et al.* 2002, Kim *et al.* 2010).

Two overlapping endothermic peaks were revealed on the heating plot of the TF specimen, indicating that reverse transformation of the alloy passes through the intermediate R-phase, which reflected the complex phase transformation behaviour tracing back to the manufacturing process. The ΔH calculated was lower for K3 than for TF (Table 1); this further suggested that higher amount of stable, work-hardened martensite (which does not undergo transformation to austenite) existed in K3 (Brantley *et al.* 2002b).

The load–deflection curves of both instruments indicated their super-elasticity at 37 °C (Fig. 2), which is attributed to a stress-induced martensitic and reverse transformation (Miura *et al.* 1986). Table 2 demonstrated that TF is more flexible than K3. Cross-sectional configuration may be the main factor affecting the bending properties of instruments (Berutti *et al.* 2003, Schäfer *et al.* 2003, Melo *et al.* 2008, Câmara *et al.* 2009) and the cross-sectional area of K3 was two times that of TF (Oh *et al.* 2010). However, other contributing factors may exist and complicate the analysis.

Particularly, the 0.5-mm deflection bending load corresponds to the elastic range and depends on the elastic moduli of the specimens, which vary amongst different crystalline phases. That of the martensite is much lower than that of austenite, with values of 28.0 GPa versus 75.0 GPa, respectively (Zinelis *et al.* 2010). On the other hand, it was reported that the elastic modulus of Nitinol, a work-hardened, nonsuperelastic NiTi wire, is $5–6 \times 10^3$ kg mm^{-2}, converted to 49.0 GPa (Miura *et al.* 1986). Thus, although TF and K3 were composed of austenite at 37 °C (Table 1), the higher amount of work-hardening in K3 could be one of the reasons for the higher load value at this point.

The super-elastic range is represented by 2.0-mm deflection bending load, which is dependent on the critical stress required to induce martensitic transformation. A previous study showed that critical stress was inversely correlated with the M_s temperature, as lower M_s impedes the phase transformation and more stress is required to induce martensitic transformation (Miyai *et al.* 2006). Thus, the lower load value for TF in this range could be explained by the higher M_s of TF compared with K3 (Table 1). The two-step transformation through an R-phase observed for TF (Fig. 1) may be another reason, because the elastic modulus of R-phase is even lower than that of martensite (Wu *et al.* 1990).

The stress hysteresis was obviously smaller for TF than for K3 (Fig. 2). The amount of stress hysteresis is determined by the interfacial energies of the phase boundaries (Xu & Müller 1991). A narrower stress hysteresis means that more austenite can be transformed during the stress-induced martensitic transformation (Liaw *et al.* 2007). This, coupled with the decreased force during the loading process, makes the insertion of TF into curved canals easier than K3.

Conclusions

The thermal behaviour and bending properties of TF NiTi endodontic instruments, manufactured by twisting, and K3 NiTi endodontic instruments, manufactured by machining, were investigated using DSC and a cantilever-bending test. Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. A_f temperatures of TF and K3 (18.88 ± 1.70 °C and 3.88 ± 3.21 °C, respectively) were lower than the body temperature of 37 °C.

2. The bending load values in elastic and super-elastic ranges were lower for TF than those of K3 at 37 °C. TF may be more flexible than K3.

3. The higher M_s temperature of TF may require the lower critical stress to induce martensitic transformation, compared with K3. Phase transformation temperature might have an impact on mechanical properties of NiTi instruments.

4. The new method of manufacturing NiTi instruments by twisting coupled with heat treatment may contribute to the superior flexibility and increased phase transformation temperatures.

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