International Endodontic Journal

doi:10.1111/j.1365-2591.2011.01891.x

Heat treatment of nickel–titanium rotary endodontic instruments: effects on bending properties and shaping abilities

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Abstract

Ebihara A, Yahata Y, Miyara K, Nakano K, Hayashi Y, Suda H. Heat treatment of nickel–titanium rotary endodontic instruments: effects on bending properties and shaping abilities. *International Endodontic Journal*, **44**, 843–849, 2011.

Aim To evaluate the bending properties and shaping abilities of nickel–titanium endodontic instruments processed by heat treatment.

Methodology K3 files were heated for 30 min at 400 °C (group 400), 450 °C (group 450) and 500 °C (group 500). Files that were not heat treated served as controls. A cantilever-bending test was used to evaluate changes in specimen flexibility caused by heat treatment. Curved root canal models were prepared. The times required for preparation, deformation and fracture were recorded. Pre- and postoperative images were superimposed. The amounts of resin removed from both the inner and the outer sides of the curvature in the apical 6 mm were determined.

Results In the cantilever-bending test, load values of the control group and group 500 were higher than those of groups 400 and 450 at the elastic range ($P < 0.05$). At the superelastic range, the bending load of the control group was the highest amongst all groups ($P < 0.05$). Regarding shaping ability, in the control group, root canals at the apex were transported more to the outer side of the curvature compared with those of all heat-treated groups ($P < 0.05$). Root canals of group 400 at 3 mm from the apex were transported less compared with those of other groups ($P < 0.05$). No significant difference was found in working time amongst the groups. In group 450, there was no plastic deformation or fracture of the file.

Conclusions Heat treatment of files might improve their flexibility, making them more effective for preparation of curved canals.

Keywords: bending property, heat treatment, K3, nickel–titanium, shaping ability.

Received 14 February 2011; accepted 12 April 2011

Introduction

Root canal instruments made of nickel–titanium (Ni–Ti) alloy exhibit greater flexibility compared with those made of other alloys, such as stainless steel (Walia *et al.* 1988). The flexibility of Ni–Ti alloy offers clinical advantages in the preparation of curved canals. Therefore, various types of Ni–Ti rotary endodontic instruments have been developed and marketed. These files differ in design, cross-sectional shape, taper and manufacturing process. Previous studies related to Ni–Ti rotary endodontic instruments have mainly focused on cutting ability (Schäfer & Florek 2003, Ayar & Love 2004, Sonntag *et al.* 2007, Shen & Haapasalo 2008), instrument fracture (Troian *et al.* 2006, Barbosa *et al.* 2008a) and cyclic fatigue (Yao *et al.* 2006, Barbosa *et al.* 2007, Ray *et al.* 2007).

On the other hand, a new generation of Ni–Ti instruments was recently introduced and evaluated:

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(i) the ProFile GT Series X, made of the new M-Wire Ni–Ti material; (ii) the Twisted File, which involves R-phase heat treatment, twisting of the metal and special surface conditioning; and (iii) the V-Taper, made of a modified Ni–Ti alloy called Endonol (Gambarini *et al.* 2008, Johnson *et al.* 2008, Alapati *et al.* 2009, Kell *et al.* 2009, Larsen *et al.* 2009, Whipple *et al.* 2009). Characteristics of these files were improved not by changing the cross-sectional shape, but by modifying the Ni–Ti alloy and its manufacturing process. Therefore, it would be interesting to examine the mechanical properties of Ni–Ti files made with a different manufacturing process, including heat treatment.

It is well known that Ni–Ti alloy has special properties such as superelasticity and shape memory (Miyazaki *et al.* 1982, Miyazaki & Otsuka 1989). Because superelasticity is caused by stress-induced martensitic transformation, it is observed at temperatures above the reverse-transformation finish point, A_f (Yoneyama *et al.* 1993). The phase transformation behaviour of Ni–Ti alloy is influenced by many factors, such as changes in composition, machining characteristics and differences in heat treatment (Thompson 2000). A few studies have examined the transformation behaviour of Ni–Ti alloy used in endodontic instruments (Kuhn & Jordan 2002, Miyai *et al.* 2006, Hayashi *et al.* 2007, Yahata *et al.* 2009, Hou *et al.* 2011, Zinelis *et al.* 2010).

The bending properties of Ni–Ti endodontic files are closely related to transformation behaviour, and it is noted that this behaviour is transformed by heat treatment (Yoneyama *et al.* 1993, Thompson 2000, Kuhn & Jordan 2002). Heat-treated Ni–Ti alloy wires reportedly had greater flexibility than that of the original alloy wire (Yoneyama *et al.* 1993). The flexibilities of four types of Ni–Ti instruments were evaluated, and it was concluded that Ni–Ti systems using less tapered and more flexible instruments, such as the S-Apex, are favourable when preparing S-shaped canals (Bonaccorso *et al.* 2009). However, the shaping ability of files with different flexibilities remains to be determined. The efficiency of these heat-treated files in root canal preparation should be evaluated because heat treatment of Ni–Ti files might improve flexibility.

The purpose of this investigation was to evaluate the bending properties of Ni–Ti rotary endodontic instruments processed by heat treatment and the shaping abilities of these files in simulated curved canals.

Materials and methods

Heat treatment

K3 files (SybronEndo, Orange, CA, USA) were used. After randomly dividing the files into four groups, they were heated in a nitrate bath (AS-140 nitric acid salt; Parker Netsushori Kogyo Co., Ltd, Tokyo, Japan) for 30 min at 400 °C (group 400), 450 °C (group 450), and 500 °C (group 500), respectively. The files were then immediately quenched in water. Files that were not heat-treated served as controls.

Cantilever-bending test

Five files with 0.30 mm tip diameters and 0.06 tapers from each group were subjected to this test. A cantilever-bending test apparatus described in previous studies (Miyai *et al.* 2006, Yahata *et al.* 2009) was used to evaluate changes in specimen flexibility caused by heat treatment. A file was first mounted on a movable stage, and the temperature of the file and apparatus was maintained at 37 °C. The cantilever-bending test was performed with a maximum deflection of 3.0 mm. The distance between the clamp edge and the file tip was 9.5 mm, and the initial loading point was 3.0 mm distant from the file tip. The bending load was measured at 0.5-mm deflection, which corresponded to the elastic range and 2.0-mm deflection to the superelastic range.

Root canal preparation

Forty standardized plastic curved root canal models (Endo Training Blocks; Dentsply Maillefer, Ballaigues, Switzerland) were randomly divided into four groups. Root canals were prepared using a Dentaport ZX (J. Morita, Kyoto, Japan) at 300 rpm by the same operator. The crown-down technique was performed using a gentle in-and-out motion. The working length was 17 mm. All canals were prepared as follows: A size 4 ProFile Orifice Shaper (Dentsply Maillefer) was used to enlarge the coronal one-third of the working length. A size 30, 0.06 taper was used to 14 mm; a size 25, 0.06 taper was used to 15 mm; and a size 30, 0.04 taper was used to 16 mm. Next, a size 25, 0.04 taper, size 30, 0.04 taper, size 25, 0.06 taper, and size 30, 0.06 taper were used to the full working length. Copious irrigation with water was performed after the use of each file. During preparation, RC-Prep (Premier, Plymouth Meeting, PA, USA) was used to fill the simulated canal.

The time required for canal preparation was recorded. The times for changes within the described instrumentation sequence and irrigation were excluded. Deformed and fractured instruments during preparation were also recorded. When instruments were fractured in the root canal, the canal was excluded and replacement canals were prepared.

Assessment of canal preparation

Images of each sample before and after instrumentation were taken by a digital microscope (VH-8000; Keyence, Osaka, Japan). A composite image of the pre- and postoperative images was produced and superimposed. The amounts of resin that were removed, which reflected the difference between the canal configuration before and after instrumentation, were determined from both the inner and the outer sides of the curvature in 3-mm steps at three levels from the apex in the apical 6 mm. Measurements of the canals were performed by a second examiner who was blinded to the experimental groups.

Analysis of data

The data of the cantilever-bending test were statistically analysed by the Tukey–Kramer test. The data of working time and canal transportation at three levels in the apical 6 mm were analysed by Fisher's test. The data of fracture and deformation was analysed by chi-square test. A significant difference was detected at a level of $P < 0.05$.

Results

Load–deflection curve

Typical load–deflection curves for each heat-treated group and the control group, indicating superelastic behaviour, are shown in Fig. 1. In all groups during the loading process, the initial portion of the load–deflection curve showed a linear relationship because of elastic deformation. Above this range, the load level became almost constant because of stress-induced martensitic transformation. During the unloading process, the load decreased rapidly and became constant owing to reverse transformation. Elastic unloading then occurred with a small permanent residual deflection.

Cantilever-bending test

Figure 2 shows the bending load at 0.5-mm deflection, corresponding to the elastic range. At 0.5-mm deflection, load values of the control group and group 500 were higher than those of groups 400 and 450 ($P < 0.05$) (Fig. 2).

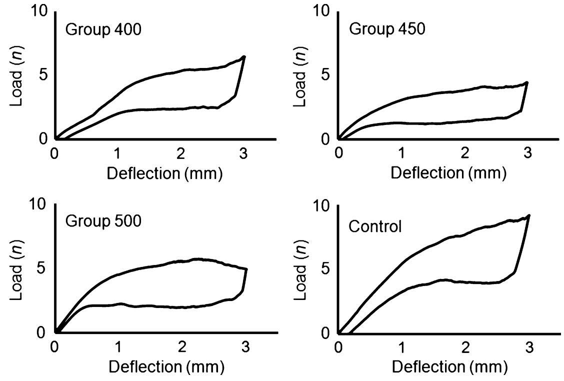

Figure 1 Typical load–deflection curves for each group.

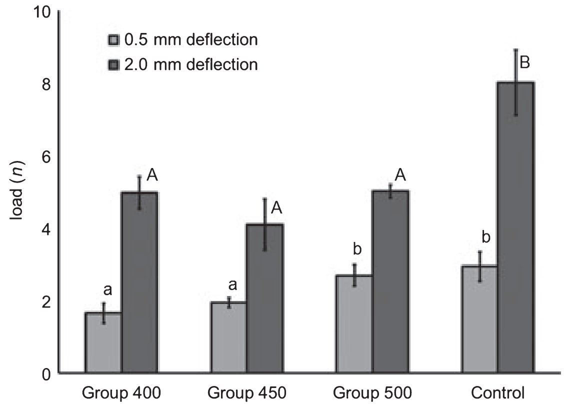

Figure 2 Bending load at 0.5 and 2.0-mm deflection for each group. The same superscript letters are not significantly different ($P > 0.05$).

Figure 2 shows the bending load at 2.0-mm deflection, corresponding to the superelastic range. At 2.0-mm deflection, the bending load values of the control group showed the highest value amongst all groups ($P < 0.05$) (Fig. 2).

Root canal preparation

Figure 3 shows a schematic drawing of the superimposed image of pre- and postoperative images. In the control group, root canals at the apex were transported more to the outer side of the curvature after preparation compared with those of all heat-treated groups ($P < 0.05$) (Fig. 4). Root canals of group 400 at 3 mm from the apex were transported less compared with

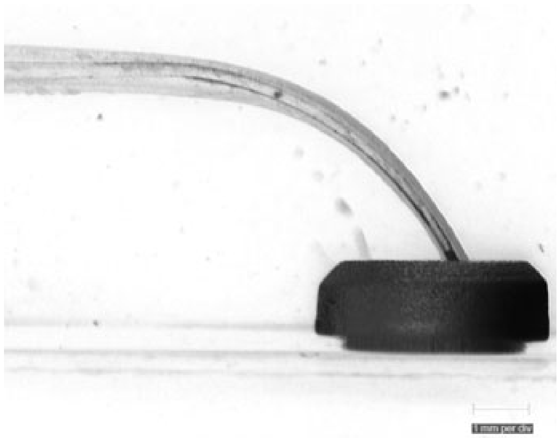

Figure 3 Superimposition of pre- and postoperative images.

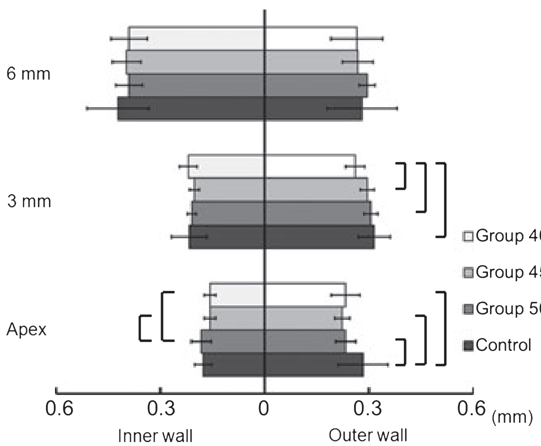

Figure 4 The amount of root canal wall cutting at the apex, 3 and 6 mm from the apex. The bars indicate significant differences between each group ($P < 0.05$).

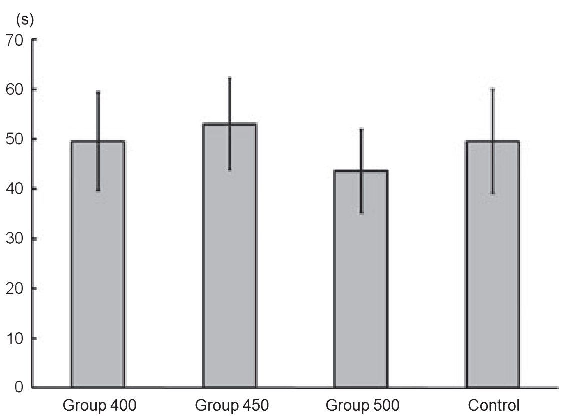

Figure 5 Working time of each group. There were no significant differences between any groups ($P > 0.05$).

those of the other groups ($P < 0.05$) (Fig. 4). At 6 mm from the apex, there were no significant differences in any of the groups (Fig. 4).

Working time

There was no significant difference in working time amongst the groups ($P > 0.05$) (Fig. 5).

Deformation and fracture

In group 450, there was no plastic deformation or fracture of files. In group 400, one file showed plastic deformation and one file fractured. In group 500, three files showed plastic deformation and one file fractured. In the control group, one file showed plastic deformation and five files fractured. There was no correlation between failure of files and instrument type ($P > 0.05$).

Discussion

Bending properties

The transformation temperature is one of the most important factors that influence the mechanical properties of Ni–Ti alloys. The phase transformation behaviour of Ni–Ti alloys can be recognized by differential scanning calorimetry (DSC) measurement (Kuhn & Jordan 2002, Miyai *et al.* 2006, Hayashi *et al.* 2007, Yahata *et al.* 2009, Hou *et al.* 2011). There are differences amongst transformation temperatures of commercial Ni–Ti rotary instruments, resulting in differences in their bending properties (Miyai *et al.* 2006). In DSC measurement, the transformation temperature of K3 instruments was reportedly significantly lower than those of EndoWave, ProFile and ProTaper instruments; and in the cantilever test, the bending load value at 3.0-mm deflection for K3 instruments was significantly higher than those of EndoWave, ProFile and ProTaper instruments (Miyai *et al.* 2006). It was also reported that all sizes and tapers of K3 files were significantly less flexible than those of FlexMaster, Hero, ProFile and Race files (Schäfer *et al.* 2003). Therefore, whether the bending value of K3 files could be decreased by heat treatment was examined in the current study. In the cantilever test, the bending loads at 0.5 and 2.0 mm corresponded to the elastic and superelastic ranges, respectively. Based on these results, heat treatment effectively decreased the bending value of K3 files at both the elastic and the superelastic ranges. In this study, the high bending property value of the no heat-treated files was probably caused by their low M_s temperature compared with those of the heat-treated groups because more stress was required to induce martensitic transformation. It was speculated that the M_s temperatures of the control group and group 500 would be similar because there was no significant difference in the elastic range between these groups. DSC measurement of heat-treated K3 files should be performed in the future to confirm changes in the phase transformation behaviour.

Heat treatment

The mechanical properties of Ni–Ti endodontic instruments are controlled by cold work and heat treatment during manufacturing (Kuhn *et al.* 2001). Thermal treatments of approximately 400 °C before machining are reportedly effective in reducing work hardening of the alloy (Kuhn & Jordan 2002). Recently, hybrid files that had undergone additional heat treatment at the tip were introduced (Hayashi *et al.* 2007). The transformation temperature was reportedly higher in the heat-treated groups than in the unheated group, and the bending load values of those files were significantly lower than those of files with no additional heat treatment in the cantilever-bending test (Hayashi *et al.* 2007). It has also been suggested that heat treatment is an important procedure that can be used to manifest the original mechanical properties of Ni–Ti alloy by releasing crystal lattice defects and contributing to changes in the phase transformation behaviour (Kuhn *et al.* 2001, Kuhn & Jordan 2002, Hayashi *et al.* 2007, Yahata *et al.* 2009).

The heat-treatment conditions used in this study were based on those of previous studies (Yoneyama *et al.* 2002, Yahata *et al.* 2009). The influence of heat-treatment temperatures below 300 °C is not sufficient to release crystal lattice defects. Recrystallization occurs above 600 °C, and superelasticity and shape memory are incomplete in this range. Therefore, heat treatment is usually performed at temperatures between 300 and 600 °C (Yahata *et al.* 2009). In the current study, heat-treatment temperatures of 400, 450 and 500 °C were selected. The influence of the heat-treatment time was reportedly less than that of the heat-treatment temperature, and longer heat treatments tended to increase the transformation temperatures (Yahata *et al.* 2009). Therefore, only one condition of heat-treatment time was adapted in the current study, and the heat-treatment period was set at 30 min. A heat-treatment temperature of 440 °C for 30 min also reportedly exhibited minimum bending load values, and it was concluded that the change in the transformation behaviour with heat treatment might have a significant effect on the mechanical properties of Ni–Ti endodontic instruments (Yahata *et al.* 2009).

Root canal preparation

Because flexibility of the files is important for the preparation of curved root canals, the cutting efficiency of heat-treated files in curved root canals was evaluated in this study. The original canal curvature should be maintained during instrumentation of root canals. Canal transportation occurs in curved canals, particularly at the outer curve of the apical portion of the canal. Superimposition of pre- and postoperative images can be applied to resin simulated root canals, and canal transportation can be evaluated at any point of the canals using PC-based measurement (Schäfer *et al.* 2003, Ayar & Love 2004, Sonntag *et al.* 2007, Bonaccorso *et al.* 2009). It was reported that K3 files prepared curved canals rapidly and with minimal transportation towards the outer aspect of the curve compared with stainless steel hand K-Flexofiles (Schäfer *et al.* 2003). It was also reported that K3 files more effectively removed outer walls of root canals compared with inner canals (Ayar & Love 2004). The present results indicate that heat treatment might improve the shaping ability of K3 files.

Use of resin simulated root canals does not reflect the action of instruments in real root canals. However, resin blocks enable comparison of the shaping abilities of different instruments. Micro CT was recently applied to evaluate the shaping ability of human root canals and canal transportation after preparation was evaluated (Gekelman *et al.* 2009, Pasternak-Júnior *et al.* 2009). The shaping ability of heat-treated files should be evaluated using human root canals in the future.

Instrument fracture and deformation

Other concerns are instrument fracture (Barbosa *et al.* 2007, 2008a) and cyclic fatigue (Yao *et al.* 2006, Ray *et al.* 2007, Gavini *et al.* 2010). The maximum torsional torque values of HERO, K3 and ProTaper files were reportedly significantly higher than those of EndoWave, ProFile and K-files (Miyai *et al.* 2006). It was speculated that K3 files had resistance to fracture because of their high torsional torque value. K3 files exhibited significantly more cycles to fracture than did the EndoSequence (Ray *et al.* 2007). K3 was also reportedly more resistant to failure than were ProFile and Race (Yao *et al.* 2006). However, fractures of K3 instruments occurred significantly more often compared with stainless steel hand K-Flexofiles (Schäfer *et al.* 2003). In the current study, there was no plastic deformation or fracture of files in group 450. Therefore, heat treatment might effectively reduce fracture of instruments. The new generation of files was recently evaluated. ProFile size 25, 0.04 files manufactured from M-Wire NiTi had significantly greater resistance to cyclic fatigue whilst maintaining comparable torsional properties (Johnson *et al.* 2008). It was also reported that ProFile GT series X made of M-Wire had a higher resistance to torsional failure after use compared with the ProFile GT (Kell *et al.* 2009). Electrochemical polishing reportedly had no influence on resistance to fracture of K3 rotary instruments (Barbosa *et al.* 2008b). In addition, ion implantation to modify mechanical properties has been attempted (Wolle *et al.* 2009, Gavini *et al.* 2010). It is important to investigate not only the cross-sectional shape of instruments, but also the mechanical properties of NiTi alloy for instrument fracture. Therefore, the influence of heat treatment on resistance to instrument fracture should be considered in the future.

Conclusions

The bending properties and shaping abilities of Ni–Ti endodontic instruments processed by heat treatment were evaluated using a cantilever-bending test and root canal preparation of curved canal models. The following conclusions were obtained.

1 Load values of the control group and group 500 were higher than those of groups 400 and 450 at the elastic range.

2 At the superelastic range, the bending load of the control group was the highest amongst all groups.

3 Root canals at the apex were transported more to the outer side of the curvature in the control group compared with those of all heat-treated groups. Root canals of group 400 at 3 mm from the apex were transported significantly less compared with those of other groups.

4 No significant difference was found in working time between heat-treated files and no heat-treated files.

5 Within the limitations of this study, the results indicate that heat treatment of files might improve their flexibility, making them effective for preparation of curved canals.

Acknowledgements

This work was supported in part by a Grant-in-Aid for Scientific Research (B) (No. 21791840 to H.Y.) from the Ministry of Education, Culture, Sports, Science and Technology of Japan and a Grant-in-Aid for Young Scientists (Start-up) (No. 19890074 to H.Y.) from the Japan Society for the Promotion of Science.

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