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ORIGINAL ARTICLE

Influence of heat treatment on cyclic fatigue and cutting efficiency of ProTaper Universal F2 instruments

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cutting efficiency;
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Abstract *Background/purpose:* Heat pretreatment can improve the cyclic fatigue life of nickel–titanium (NiTi) instruments. This study evaluated the effects of two different heat treatments on the cyclic fatigue resistance and cutting efficiency of ProTaper Universal F2 files.
Materials and methods: The files were divided into three groups: no treatment (control), heat treatment at 400°C (HT400) and heat treatment at 600°C (HT600). The phase transformation of the files was evaluated by differential scanning calorimetry. In cyclic fatigue tests, the differences in file performance in four simulated canals among the three groups were assessed. The cutting efficiency was tested at four cutting portions (3 mm, 6 mm, 9 mm, and 12 mm) from the tip of the file.
Results: Differential scanning calorimetry showed a prolonged phase transformation of the files only after 600°C treatment. At 3 mm cutting portion, 400°C heat-treated files had significantly better cutting ability than those in the control group. However, the files in the HT600 group had significantly lower cutting efficiency than those in the other two groups at the four tested positions. In the cyclic fatigue test, fatigue lives of the files after 400°C and 600°C

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treatment were prolonged from 2.1 to 2.8 times and from 1.7 to 5.5 times, respectively.
Conclusion: Although 600°C treatment increased resistance to cyclic fatigue, it reduced the cutting efficiency of the files. The 400°C treatment maintained the cutting ability and prolonged the cyclic fatigue life of the files. Therefore, for clinical use of ProTaper Universal F2 files, 400°C pretreatment is a better choice than 600°C pretreatment.

Introduction

Nickel–titanium (NiTi) alloy has been popular in endodontic treatment due to its superelasticity. NiTi rotary instruments facilitate intracanal instrumentation and prevent iatrogenic deviation of the canal.[1] Despite their superior properties, unexpected fatigue fractures of the instruments occur as the instruments operate under alternating tensile and compressive stress during operation.[2–4] Fragmentation of the instrument may affect the outcome of endodontic treatment and embarrass the clinics.[5]

Modifications on geometric design,[6–8] surface treatment,[9–11] and manufacturing process have been proposed for enhancing the cutting efficiency and fatigue resistance of NiTi rotary instruments.[12,13] Zinelis et al[14] showed that heat treatment on the apical 5 mm position of a NiTi rotary instrument can extend its fatigue life due to reducing residual strain. Furthermore, NiTi files have more resistance to cyclic fatigue after heat treatment at 430°C.[14] Besides, heat treatment of NiTi rotary instruments also results in superior elastic behavior and provides better shaping ability.[15] Heat treatments such as tempering, quenching and annealing have been common industrial practices to improve the mechanical properties of metallic components. Annealing can relieve the residual stress in instruments and improve their ductility, toughness, and superelasticity.[16,17] Previous studies have shown that phase transformation behavior changes under annealing treatment of NiTi alloy. The strength of NiTi alloy decreases as the annealing temperature increases.[18] In heat treatment at up to 600°C, the NiTi alloy undergoes martensitic and R-phase formation.[19] These can improve mechanical behavior of NiTi alloys. Even though this process can improve the mechanical behavior of NiTi alloy, the effect of heat treatment on the cutting efficiency of NiTi instruments is still unclear.

In this study, the effects of different heat treatments on the cyclic fatigue resistance and cutting ability of NiTi rotary instruments were investigated by measuring their phase transformation points through heating processes. The results of this study could provide intrinsic characteristics of endodontic NiTi instruments for evaluating their potential for future clinical applications.

Materials and methods

Heat treatment for ProTaper Universal F2 instruments

The ProTaper Universal F2 instruments (Dentsply Tulsa Dental, Tulsa, OK, USA) were chosen and randomly divided into three groups: no treatment (control), heat treatment at 400°C (HT400), and heat treatment at 600°C (HT600). The files in the latter two groups were heat-treated in a nitrate bath at 400°C and 600°C, respectively. The instruments were brought to within 50°C of their respective treatment temperatures with a rapid temperature rise of 10°C/min. The temperature rise was then reduced to 5°C/min until the designated temperature was reached. Thereafter, the instruments were kept at that temperature for 15 minutes before they were furnace cooled. The nitrate bath temperature in this study was controlled by a digital proportional–integral–derivative (PID) controller (TAIE FY900, Taipei, Taiwan).

Assessment of phase transformation

To investigate the effect of heat treatment on the phase transformation behavior of the instruments, differential scanning calorimetry (DSC) measurement was made using the LT-Modulate DSC 2920 (TA Instruments, New Castle, DE, USA). The specimens were cut into 2-mm segments and ultrasonically cleaned in acetone for 5 minutes. For each group, 20 g of the cut specimen was put into the DSC cell. Scanning was made in the temperature range from −80°C to 100°C in an argon atmosphere. During the heating and cooling processes, the rate of temperature change was set at 5°C/min. The heating and cooling processes for each group were repeated twice. Temperature corresponding to martensitic transformation starting point (M_s), martensitic transformation finishing point (M_f), reverse transformation starting point (A_s), reverse transformation finishing point (A_f), and the enthalpy of phase transformation were recorded.

Cyclic fatigue test

Cyclic fatigue testing in artificial canals was performed with an electric motor (TCM endo III, Sybron Endo, Glendora, CA). The simulated canals were formed by a steel cylinder of radius (R) and a block with matching circular arc (θ).[20] The simulated canals were set as R10mmθ20°, R7.5mmθ40°, R7.5mmθ60°, and R5mmθ60°. The rotation speed was set at 350 rpm without torque control. The fracture time was recorded for each file in seconds and repeated five times ($n = 5$). The corresponding fatigue life in number of cycles was then accurately calculated.

Cutting efficiency test

The device for cutting efficiency evaluation was composed of two electric motors: one to rotate the instrument, and the other to rotate two cylindrical acrylic blocks to be cut. The two acrylic blocks were pressed against the instrument with a spring loaded device to allow the contact force to be adjusted and held constant (Figure 1). When the file rotated, the acrylic blocks also counter-rotated to expose a fresh surface for cutting. A spring force of 10 N to press against the instrument was used.

Five files in each group were used for the cutting efficiency test. For each file, four cutting regions, namely, 3 mm, 6 mm, 9 mm, and 12 mm from the tip of the file were tested. The files were rotated at 300 rpm and the cutting time for each test session was 40 seconds. Before and after each cutting session, the resin blocks were ultrasonically cleaned in a 75% alcohol bath, and then thoroughly dried and weighed with a microscale AUW220D (Shimadzu Corporation, Kyoto, Japan) to a resolution of 0.1 mg. The cutting efficiency of the instrument was quantified by the weight loss during the test.

The results from cyclic fatigue tests and cutting efficiency tests were statistically analyzed by one-way analysis of variance and *post hoc* Bonferroni test at P = 0.05 with statistical analysis software (SPSS for Windows version 16; Chicago, IL, USA).

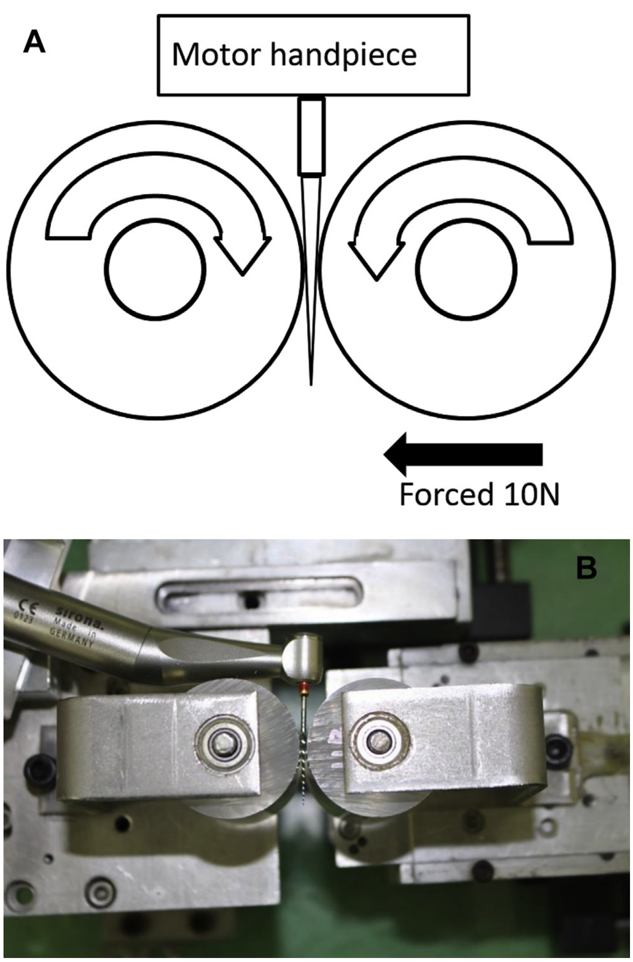

Figure 1 Component of cutting efficiency test device. (A) Illustration of the device. There were two counter rotated acrylic blocks that contacted the file with a force of 10 N. The file was operated by motor. (B) Photograph of the device.

Results

Behavior of phase transformation

Figure 2 shows the DSC curves for the control, HT400 and HT600 groups. The phase transformation temperatures (A_s, A_f, M_s and M_f) for the files in the control group were −5°C, 26°C, 21°C, and −14°C, respectively. The files in the HT400 group showed elevated phase transformation temperatures of 34.7°C, 50°C, 44°C, and 28°C, respectively. Conversely, the files in the HT600 group gave depressed phase transformation temperature of −14°C, 35°C, 0°C, and −70°C, respectively. When the DSC curves were compared among different groups, the control and HT400 groups showed similar behavior with a single peak in the heating and cooling process. The curve of the HT600 group showed a prolonged transformation area in both heating and cooling processes. Furthermore, the enthalpy of phase transformation of the files in the HT600 and HT400 groups increased, compared with that in the control group.

Cyclic fatigue test

In cyclic fatigue tests, there were significant differences in the cyclic fatigue life of the files between any two of the control and two different experimental groups with files testing in the four simulated canals, except the data between the control and HT400 groups in canals of R7.5mmθ60°, and the data between HT400 and HT600 groups in canals of R7.5mmθ40° (Table 1). The HT600 group showed the most superior fatigue resistance in the simulated canals except in those of R7.5mmθ40°. In R7.5mmθ40° simulated canals, files in the HT400 group showed marginally better fatigue life than those in the HT600 group. The files in the HT400 group had more prolonged fatigue lives from 2.1× to 2.8× than those in the control group. In addition, the files in the HT600 group also had more prolonged fatigue lives from 1.7× to 5.5× than those in the control group.

Cutting efficiency test

Comparisons of the cutting efficiency at the four different cutting portions of the files in the control and two different experimental groups are shown in Table 2. Except the cutting efficiency at the 6-mm cutting portion of the files between the control and HT400 groups, there were significant differences in the cutting efficiency at the four different cutting portions between any two of the groups. In general, the files in the control group had better cutting efficiency than the files in the other two groups. However, the files in the HT400 group had significantly better cutting efficiency than the files in the control group at the 3 mm cutting region. Furthermore, the files in the HT600 group had significantly lower cutting efficiency than the files in the control and HT400 groups at all four cutting positions. The degradation in cutting efficiency ranged from 10.1% to 27.0%.

Discussion

For clinical root canal preparation, NiTi rotary files are capable of maintaining canal centrality and building an

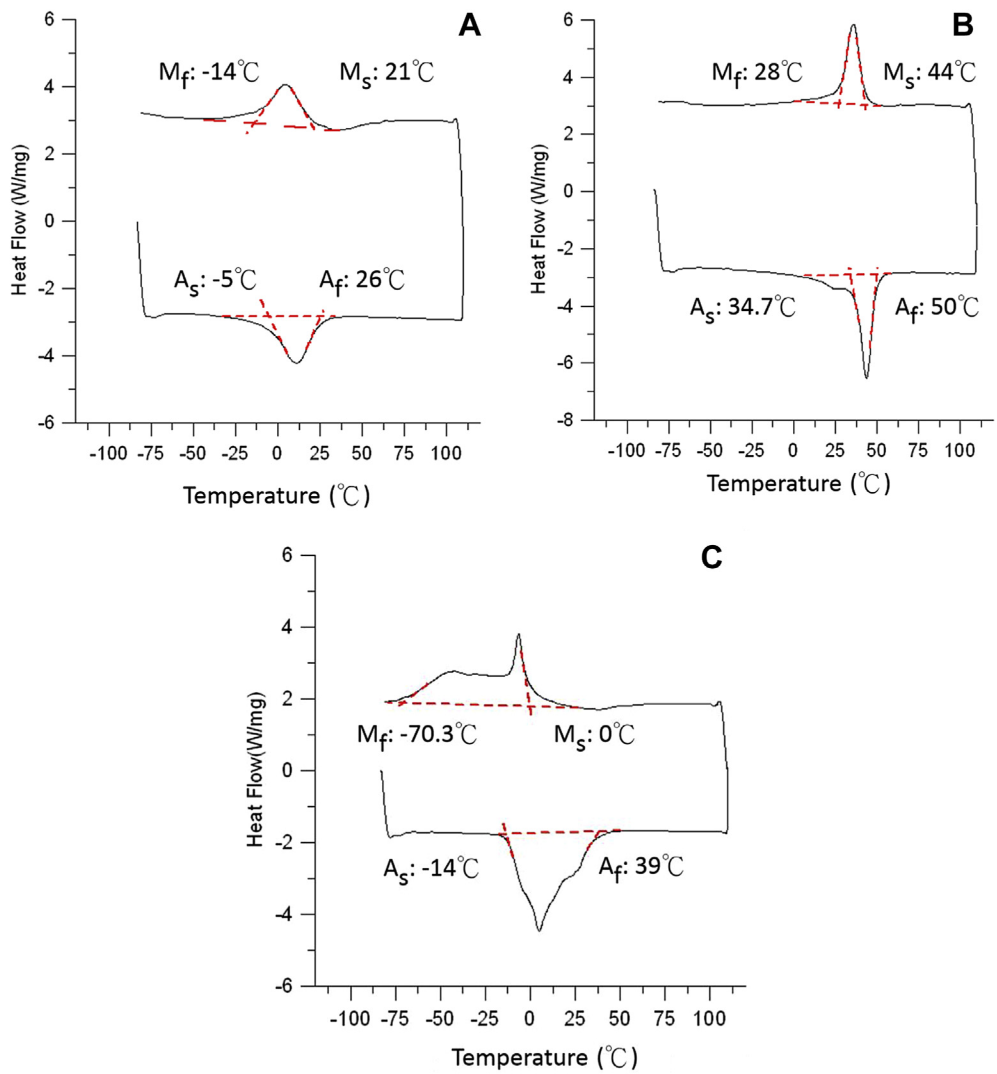

Figure 2 DSC results. (A) Control group: normal behavior of ProTaper Universal F2; (B) HT400 group: there was a single sharper and higher peak over the heating and cooling cycles; and (C) HT600 group: there was one peak combined with one plateau over the test. DSC = differential scanning calorimetry; HT400 = heat treatment at 400°C; HT600 = heat treatment at 600°C.

ideal 3D shape for obturation with reduced preparation time.[1] However, the occurrence of unpredictable fractures may influence the therapeutic outcomes and even further lead to poor prognosis.[2,3] In order to prevent fracture of the instruments, there have been many studies focused on the cyclic fatigue lives of NiTi instruments. A new NiTi alloy called M-wire and new manufacturing process for Twisted File were developed to achieve better cyclic fatigue lives and cutting ability.[21] Besides, the mechanical strength of the fabricated NiTi files can be further improved by additional heat treatment within a specific temperature region. A previous study found that heat treatment of NiTi alloy below 300°C was not enough to achieve a better cyclic fatigue life due to the remaining crystal lattice defect.[22]

Table 1 Cyclic fatigue lives of the files in the control and two experimental groups with files testing in the four different simulated canals.

	R10mmθ20°		R7.5mmθ40°		R7.5mmθ60°		R5mmθ60°	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Control	9953[a]	2860.9	1325.3[a]	215.2	513.6[a]	180.1	562[a]	129.1
HT400	27668.8[b]	566.1	3323.8[b]	262.3	1233.4[a]	284.7	1186.6[b]	117.8
HT600	38406.8[c]	8439.0	2855.3[b]	315.6	2680.4[b]	382.2	2142.4[c]	208.1

Different superscript letters indicate a significant difference among groups in each simulated canal.
HT400 = heat treatment at 400°C; HT600 = heat treatment at 600°C; SD = standard deviation.

Table 2 Weight loss (mg) at the four cutting portions of the files in the control and two different experimental groups.

	3 mm		6 mm		9 mm		12 mm	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Control	18.09^a	0.23	25.82^a	1.05	34.27^a	0.28	38.66^a	0.30
HT400	18.63^b	0.06	26.13^a	0.21	31.68^b	0.24	35.30^b	0.19
HT600	16.28^c	0.11	21.07^b	0.18	25.23^c	0.22	28.21^c	0.08

Different superscript letters indicate a significant difference among groups in each simulated canal.
HT400 = heat treatment at 400°C; HT600 = heat treatment at 600°C; SD = standard deviation.

Instead, another study suggested that cyclic fatigue life is obviously prolonged after heat treatment of 400–450°C.[14] It has also been reported that heat treatment above 600°C can induce recrystallization of NiTi alloy and thus decrease surface hardness.[19] It is clear that the heating temperature is an essential factor closely related to the performance of NiTi alloy. However, the effects of heat treatment with a temperature between 400°C and 600°C on NiTi files is still unclear. In the current study, both 400°C and 600°C heat treatments were evaluated for their effects on the cyclic fatigue life and cutting ability of NiTi files.

The DSC results of this study showed that A_f of the files in the control group was lower than room temperature (27°C). This finding indicates that the instrument is in the complete austenite phase. In contrast, the files in the HT400 group showed higher A_s and M_f values than room temperature, suggesting that the instruments were in the complete martensite phase. Besides, the room temperature fell between the A_s and A_f values of the files in the HT600 group; this implies that the instruments were in a mixture of austenite and martensite phases. Since the superelastic behavior and shape memory of NiTi alloy resulted from the strain induced by the phase transformation between martensite and austenite phases,[23] the files in the HT400 group had better cyclic fatigue resistance than those in the control group in all four canals tested. In fact, our result was comparable to that reported in a previous study that tested the effect of 430°C heat treatment on NiTi instuments.[24] The phenomena may be attributed to the predominant martensite phase of the files in the HT400 group at room temperature. In contrast, the files in the control group were predominantly in the austenite phase, which is known to have inferior fatigue resistance than the files in the martensite phase.[14] In contrast, heat treatment at 600°C brought A_f down to −14°C and thus increased the two-phase coexistence range. The enthalpy of the phase transformation increased up to three times higher than that in the control group. Sadrnezhaad and Mirabolghasemi[19] reported that NiTi alloy recrystallizes, thus resulting in a decrease in its hardness after heat treatment at 600°C. Also, R-phase formation, martensitic crystal transformation, recovery of microstructure, and order–disorder transformation have been proposed to be the reasons for mechanical behavior changes.[19] Therefore, the superior cyclic fatigue resistance of files in the HT600 group compared with those of the files in the control and HT400 groups may be attributed to the aforementioned factors.

Cutting efficiency has been measured with several different methods, including weight loss,[25,26] debris generated after instrumentation,[27] measuring the maximum penetration depth into the lumen,[28] preparation time,[29,30] and cutting depth in the resin block.[31] Among different evaluation methods, weight loss gives the most reliable quantitative measurement. Since the groove of the canal cut was found to increase its size during the course of testing, the contact area and position as well as the contact pressure and angle would therefore change accordingly. This phenomenon is further aggravated as most of the instruments have a tapered geometry. Although previous methods for cutting efficiency investigation might closely mimic the action of root canal treatment clinically, they did not present an equal basis for comparing the cutting capability of files with different designs and different cutting portions on the same file.

Furthermore, most of the prior studies evaluated the instrument as a whole and could not evaluate the specific region of the instrument. Rubini introduced the method to evaluate the specific cutting region.[31] However, bending of the file during the test that causes different pressing contact forces may result in variable outcomes.

In this work, the contact between the instrument and the acrylic cylinders occupied a small length of the instrument, thus it is possible to compare the cutting efficiency of different portions of the same instrument without inducing unwanted bending strain. Moreover, the rotating blocks presented a fresh surface for cutting, maintaining the same relative cutting angle and contact pressure throughout the whole cutting period. Thus, the current method provided a fair comparison. Furthermore, cutting of the two acrylic blocks in the same session represented two independent cutting tests, giving two weight losses, which allow the reproducibility of the results to be assessed. The weight loss from both blocks demonstrated that reliable cutting efficiency data from the coronal to apical portions can be obtained.

The inferior cutting capability of the file in the HT600 group was probably associated with the lower hardness of the files after the 600°C heat treatment.[9,19] The similar cutting efficiency of the files in both control and HT400 groups at the 3 mm and 6 mm cutting portions has useful clinical implications because it indicates that 400°C heat treatment greatly improved cyclic fatigue resistance without sacrificing cutting efficiency. The slightly decreased cutting efficiency of the files in the HT400 group at the 9 mm and 12 mm cutting portions was not a great concern as these portions of the file were seldom used clinically.

In conclusion, the performance of ProTaper Universal files in cyclic fatigue life and cutting efficiency tests was modified through heat treatment. For the files in the HT400 group, the cutting efficiency was maintained as their cyclic fatigue life was prolonged compared with the files in the control group. In contrast, the cutting efficiency of the files in the HT600 group was decreased despite their superior cyclic fatigue life compared with the files in the other two groups. Considering the clinical performance of NiTi instruments, the 400°C heat treatment is better than the 600°C heat treatment.

Conflicts of interest

The authors have no conflicts of interest relevant to this article.

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