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ORIGINAL ARTICLE

Effect from the sequence of heat treatment and grinding on the mechanical properties of nickel–titanium rotary instruments

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Abstract

This study aimed to evaluate how the sequence of heat treatment and grinding affects the mechanical properties of nickel–titanium rotary instruments. ProTaper Gold (PTG) and three experimental FQ files with identical designs, but different manufacturing sequences, were tested. FQhg was heat-treated before grinding, FQgh was ground before heat treatment, and FQnh was not heat-treated. Cyclic fatigue resistance, torsional resistance, and bending stiffness were measured ($n = 15$). Scanning electron microscopy and differential scanning calorimetry (DSC) were performed. Statistical analysis was conducted using one-way ANOVA and Tukey's test at a 95% significance level. FQhg showed the highest cyclic fatigue resistance, followed by FQgh, FQnh, and PTG ($p < 0.05$), while no significant differences were found in torsional strength, toughness, or distortion angle among the FQ groups. PTG showed higher torsional strength and bending stiffness, likely due to its different cross-sectional design and taper. DSC analysis revealed minor differences in austenite transformation temperatures between FQhg and FQgh. The sequence of heat treatment and grinding significantly influenced cyclic fatigue resistance, with post-heat-treatment grinding (FQhg) providing superior performance; however, its impact on other mechanical properties was limited. The clinical relevance of processing sequence appears minimal compared to factors such as cross-sectional design and heat treatment.

KEYWORDS

bending stiffness, cyclic fatigue resistance, grinding, manufacturing process, heat treatment, torsional resistance

INTRODUCTION

The main purpose of root canal treatment is to remove infected or dead pulp tissue and eliminate bacteria while sealing the root canal properly [1]. To achieve this, effective mechanical instrumentation is essential for shaping the root canal, allowing for thorough disinfection and optimal obturation [2]. Stainless steel (SS) files were introduced for this purpose and have long been used in endodontics due to their strength and cutting ability. However, their inherent rigidity often leads to procedural complications such as canal transportation, ledging, and apical blockage, particularly in curved canals [3]. To overcome these limitations, nickel–titanium (NiTi) rotary instruments were developed. Compared to SS files, NiTi files offer superior flexibility, greater resistance to cyclic fatigue, and improved canal shaping efficiency, making them more effective in preserving the original canal anatomy [4].

Despite these benefits, NiTi files are prone to fracture, which remains a major concern in clinical practice. File separation can occur due to two primary mechanisms: cyclic fatigue and torsional failure [5]. Cyclic fatigue occurs when the instrument rotates in curved canals. During each rotation, alternating compressive and tensile stresses are generated at the point of maximum curvature, leading to the accumulation of microcracks and eventual fracture [6]. In contrast, torsional failure results from the file tip binding within the canal while the shank continues to rotate, producing excessive torsional stress that exceeds the elastic limit of the alloy. Both failure modes are clinically significant, as they may compromise root canal treatment outcomes and complicate further procedures [7].

To enhance resistance to cyclic fatigue and torsional failure, various strategies have been adopted by manufacturers, such as altering the alloy's phase transformation characteristics through heat treatment [8], modifying the cross-sectional design [9], and reducing machining-induced surface irregularities through electropolishing [10]. Among these, heat treatment has been widely used to alter the microstructure and mechanical properties of NiTi files, thereby improving their durability. Heat-treated NiTi files, including those utilizing controlled memory (CM)-wire, gold-wire, and blue-wire technologies, exhibit significantly improved flexibility and cyclic fatigue resistance compared to conventional non-treated or M-wire counterparts [11, 12]. These advanced heat treatments modify the phase transformation behavior, providing controlled memory properties for better adaptation to severely curved canals and stabilizing martensitic phases for enhanced durability even at body temperature [13].

ProTaper Gold (PTG; Dentsply Sirona) features a proprietary gold heat treatment process that enhances flexibility and cyclic fatigue resistance and has a convex triangular cross-sectional design [14]. The system consists of shaping and finishing files with progressive tapering designs, allowing efficient dentin removal while maintaining structural integrity. The unique metallurgy provides enhanced flexibility, facilitating better adaptation to curved canals and reducing procedural errors. The FQ file system (Komet Dental) is a heat-treated NiTi rotary instrument developed to enhance shaping efficiency and safety during root canal preparation. It features a fixed 6% taper, a non-cutting guiding tip, and a distinctive double-S cross-sectional geometry designed to improve cutting action and facilitate debris removal while reducing torsional stress.

Recently introduced NiTi rotary instruments are typically manufactured through a combination of machining and heat treatment, both of which influence their mechanical properties. The grinding process plays a fundamental role in shaping key geometric features such as taper, cross-sectional design, and tip configuration, which directly influence the instrument's clinical performance [15]. However, machining inevitably produces surface irregularities such as micro-notches and machining grooves that can act as stress concentrators and serve as initiation sites for crack propagation under cyclic or torsional loading [16]. Heat treatment techniques have been developed to modify the alloy's phase transformation behavior, relieve residual stress, and reduce the negative effects of surface defects. Heat treatment applied after machining may help mitigate the microstructural damage introduced during grinding by stabilizing the alloy structure [9]. In contrast, when machining is conducted after heat treatment, the mechanical benefits of thermal processing may be compromised due to the reintroduction of surface flaws [15]. The mechanical performance of NiTi files may depend on the sequence of machining and heat treatment during manufacturing. Despite the importance of both processes, there is limited research examining how their order affects the fracture resistance and durability of these instruments. A clearer understanding of this relationship is essential for optimizing file design and ensuring clinical reliability.

The influence of the processing sequence on the physical properties of NiTi instruments, particularly the order of grinding and heat treatment, has not been thoroughly investigated. Therefore, this study aims to evaluate the impact of the sequence of heat treatment and grinding processes on the mechanical properties of NiTi files. The null hypothesis of this study was that the sequence of heat treatment and grinding processes would not affect the mechanical properties of NiTi files.

MATERIAL AND METHODS

PTG and three experimental FQ groups (FQhg, FQgh, and FQnh) of NiTi rotary files were selected for this study. The manufacturer specially produced the FQ control groups used in this study at our request to match the experimental conditions. The experimental groups were defined based on their manufacturing processes as follows: FQhg was manufactured with heat treatment followed by a grinding process; FQgh underwent a grinding process first, followed by heat treatment; and FQnh was manufactured without heat treatment (Figure 1). PTG was chosen as a widely used commercial reference with a different heat treatment and geometry to assess sequence effects relative to variations in heat treatment type and design, enabling insights beyond sequence alone. The size and taper of PTG F2 were ISO #25 and 0.08 taper in apical 3 mm), and FQhg, FQgh, and FQnh were #25 and 0.06 taper. All files were used according to the manufacturers' instructions.

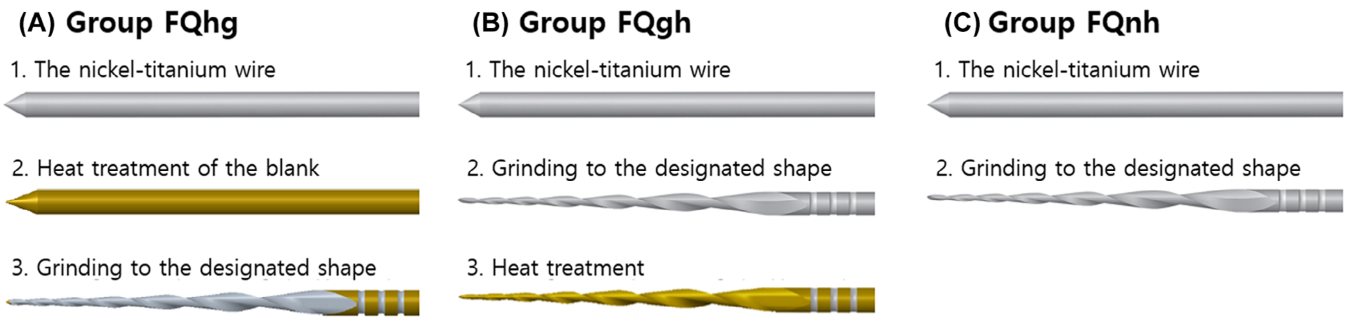

FIGURE 1 Manufacturing process of the FQ control groups. (A) FQhg was manufactured with heat treatment followed by a grinding process. (B) FQgh underwent a grinding process first, followed by heat treatment. (C) FQnh was manufactured without heat treatment.

Prior to the experiment, all instruments were examined under a microscope (Zeiss Pico; Carl Zeiss MediTex). Instruments exhibiting any visible defects or irregularities were discarded.

Sample size calculation

Sample size calculation was performed using G*Power software (version 3.1.9.7; Heinrich Heine University) with an effect size of 0.8, a significance level (α) of 0.05, and a power of 0.95. The indicated total sample size was 32 samples for each test, but to enhance statistical power and account for possible defective instruments, 15 new files were included in each of the 4 groups [17].

Cyclic fatigue resistance

Cyclic fatigue resistance was evaluated using a custom-made device, EndoC (DMJ System). It was a repeatable simulation in curved canal to confine the rotating instrument. This artificial canal block was made of tempered steel with 6 mm radius and 45° of curvature measured by the method of Schneider [18]. Synthetic oil (WD-40; WD-40 Company) was sprayed into the metal canal space to reduce friction between the instrument and the walls of the metal canal. The instruments from each subgroup ($n = 15$) were rotated at 300 rpm in pecking movement with a dynamic 4 mm up-and-down motion. Temperature was standardized at 37°C to simulate body conditions, monitored with an electric thermometer. Because the rotation speed and number of cycles to failure (NCF) are mathematically proportioned and/or the speed may have an influence on the NCF, the fatigue condition was given with the minimum recommendation speed of the tested file systems to make a fair condition. Once the instrument fracture was detected visually and audibly, the time for fracture was recorded with a chronometer. The NCF for each instrument was calculated by multiplying the total time (seconds) to fracture by the rotation rate (5 revolutions per second, 300 rpm). The length of the separated file tip was measured using a digital microcaliper (Mitutoyo).

Torsional resistance

Torsional resistance was evaluated for four groups of instruments ($n = 15$ per group) using a custom-made device, AEndoS (DMJ System) designed to measure the maximum torsional load (N·cm) until fracture. The apical 3 mm of each file was securely clamped between two copper plates, and the instruments were rotated in a clockwise direction at a speed of 2 rpm until fracture occurred. Throughout the rotation, both the applied torque (N·cm) and the distortion angle (°) were continuously recorded. Toughness was calculated as the area under the torque–distortion angle curve using Origin v6.0 Professional software (Microcal Software Inc).

Bending stiffness

Bending stiffness was assessed using a custom device (AEndoS) in accordance with ANSI/ADA Specification No. 28 and ISO 3630–1:2008. Each instrument ($n = 15$) was fixed 3 mm from the tip and bent to 45° relative to its long axis. The resulting bending moment was measured using the load cell integrated into the device. If the instrument did not return to its original shape after deflection, the residual angle was defined as the angle between the deflected and initial positions. In this measurement, the distance "a" was kept constant, while "b" was measured using a microcaliper. The residual angle (θ) was calculated using the formula: $\theta = \arctan$ (b/a).

Scanning electron microscope evaluation

The fractured fragments from the experimental groups were examined using a scanning electron microscope (SEM) (SU 5000; HITACHI). Five fractured fragments were randomly selected from each group and observed at various magnifications to analyze the surface morphology of the fractured instruments.

Differential scanning calorimetry

Differential scanning calorimetry (DSC) was used to evaluate the phase transformation behavior of the NiTi instruments, with emphasis on the austenitic transformation. This technique records the heat flow associated with phase transitions as a material is heated and cooled at a controlled rate, producing thermograms that reflect transformation events.

In this study, five specimens from each group were tested using a DSC device (DSC 25, TA Instruments) under a thermal range of –60°C to 100°C in a controlled atmosphere. This temperature span was selected to ensure full transformation beyond the austenite finish point, based on prior experimental data. Heating and cooling profiles were automatically generated to assess transformation temperatures and enthalpy. The start and finish temperatures of each phase change were identified from the intersection of tangents drawn to the thermal peaks and the baseline. These were reported as M_s (martensite start), M_p (martensite peak), M_f (martensite finish), As (austenite start), A_p (austenite peak), and A_f (austenite finish), following standard nomenclature.

Statistical analysis

The homogeneity of variances was assessed using Levene's test, and the normality of data distribution was verified using the Shapiro–Wilk test. Since the data were normally distributed, one-way analysis of variance (ANOVA) and Tukey's HSD test were performed to evaluate differences among groups. A significance level of $p < 0.05$ was applied. Statistical analysis was conducted using SPSS software (version 26; IBM Corp.).

RESULTS

Table 1 shows the results of mechanical testing for each group, including cyclic fatigue resistance, torsional resistance, and bending stiffness. FQhg showed the highest NCF, and FQnh exhibited the lowest among the groups; however, the difference between FQnh and PTG was not statistically significant ($p > 0.05$). The mean length of the separated file fragments was shortest in PTG, with no significant difference among FQ groups.

In torsional resistance, PTG had significantly higher ultimate strength than all FQ groups ($p < 0.05$), while no differences were found among the FQ groups. All FQ groups exhibited greater distortion angles than PTG ($p < 0.05$), with no differences among the FQ groups. Toughness values showed no differences across groups.

For bending stiffness, PTG showed the highest residual angle values compared to all FQ groups ($p < 0.05$), while no differences were found among the FQ groups. Bending stiffness was the highest in PTG, followed by FQnh, and lowest in FQhg and FQgh. Statistically significant differences were observed among all groups ($p < 0.05$).

After the cyclic fatigue resistance test, SEM analysis showed one or more crack initiation origins and a distinct fatigue zone in the fractured fragments (Figure 2A,D,G,J). No distinct morphological changes were observed on the lateral aspects; however, pronounced machining grooves were frequently observed in the PTG group (Figure 2C,F,I,L). Following the torsional resistance test, SEM images of the fractured fragments revealed typical concentric abrasion marks (Figure 3A,D,G,J) and dimples (Figure 3B,E,H,K) in the cross-sectional views. Distortion was observed on the lateral aspects of the specimens (Figure 3C,F,I,L).

DSC analysis revealed distinct differences in phase transformation behavior among the four NiTi file systems. PTG exhibited the highest transformation temperatures among all groups, with an austenite start temperature (A_s) of 38.63°C, an austenite peak (A_p) of 43.09°C, and an austenite finish (A_f) of 53.13°C (Figure 4A). In FQhg, A_s, A_p, and A_f were 27.77°C, 36.94°C, and 46.71°C, respectively (Figure 4B). FQgh showed slightly lower transformation temperatures, with A_s, A_p, and A_f measured at 31.96°C, 36.37°C, and 40.73°C, respectively (Figure 3C). In FQnh, which received no heat treatment, A_s, A_p, and A_f were substantially lower at 3.94°C, 13.18°C, and 24.67°C, respectively, indicating a predominantly austenitic phase under clinical conditions (Figure 4D).

DISCUSSION

Heat treatment is commonly used to modify the physical and mechanical properties of NiTi rotary instruments by altering their phase transformation characteristics [5]. The thermal processing conditions influence the phase present at clinical temperatures, thereby affecting the instrument's flexibility and fatigue resistance. While the individual effects of heat treatment and machining have been widely studied [19, 20], the impact of their application sequence remains unclear. To address this, the present study evaluated how the order of grinding and heat treatment affects the mechanical and thermal properties of NiTi rotary instruments. Three custom-fabricated FQ files with identical design and geometry were tested, differing only in the processing sequence. A commercially available heat-treated instrument, PTG, selected as the control. All NiTi files used in this study share the same alloy

TABLE 1 Mechanical properties of ProTaper Gold and FQ Instruments (mean ± standard deviation).

System	Cyclic fatigue resistance: Number of cycles to failure (NCF)	Cyclic fatigue resistance: Fracture fragment length (mm)	Torsional resistance: Ultimate strength (N·cm)	Torsional resistance: Distortion angle (°)	Torsional resistance: Toughness (° N·cm)	Bending stiffness: Residual angle (°)	Bending stiffness: Bending stiffness (N·cm)
PTG	877 ± 95.97[c]	3.24 ± 1.15[b]	1.56 ± 0.17[a]	391.38 ± 28.12[b]	411.58 ± 40.65[a]	15.34 ± 1.30[a]	0.96 ± 0.09[a]
FQhg	1181 ± 119.18[a]	4.05 ± 0.53[a]	1.17 ± 0.12[b]	525.36 ± 43.99[a]	408.51 ± 56.08[a]	13.52 ± 1.53[b]	0.64 ± 0.08[c]
FQgh	1033 ± 101.12[b]	4.41 ± 0.65[a]	1.16 ± 0.09[b]	535.49 ± 38.39[a]	401.93 ± 41.24[a]	13.25 ± 1.97[b]	0.60 ± 0.11[c]
FQnh	866 ± 139.7[c]	4.77 ± 0.66[a]	1.26 ± 0.07[b]	501.26 ± 52.37[a]	439.72 ± 44.15[a]	12.47 ± 0.73[b]	0.84 ± 0.08[b]

Note: Values in each vertical column marked with different superscript alphabets indicate significant differences ($p < 0.05$).

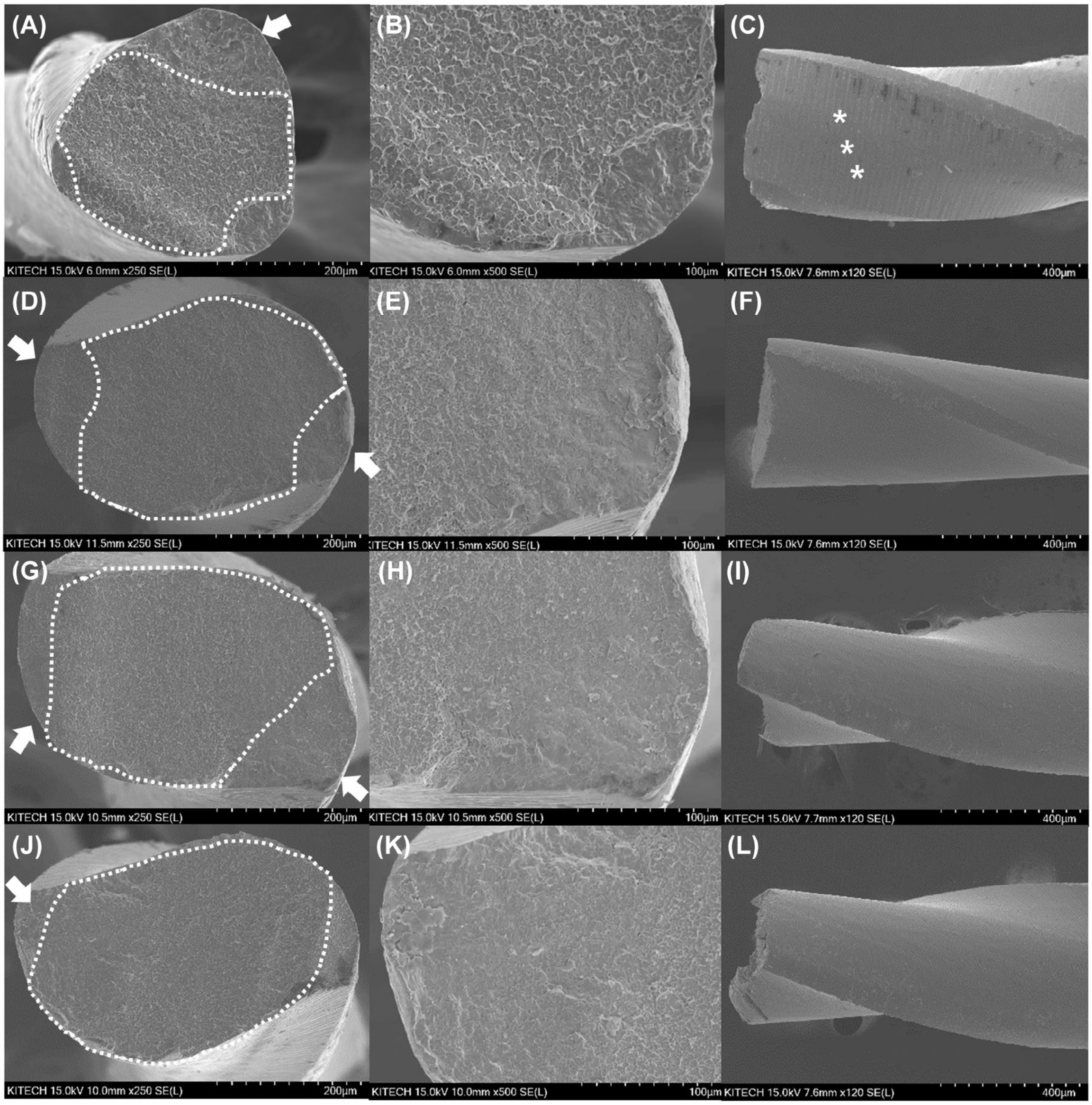

FIGURE 2 Scanning electron microscope images of separated file fragments after the cyclic fatigue resistance test. PTG: A–C; FQhg: D–F; FQgh: G–I; FQnh: J–L. Cross-sectional views (A, D, G, J) at ×250, enlarged cross-sectional views (B, E, H, K) at ×500, and lateral views (C, F, I, L) at ×120 are shown. In the cross-sectional views (A, D, G, J), white arrows indicate crack initiation origins, and dotted lines outline the fatigue zones. Asterisks indicate numerous machining grooves.

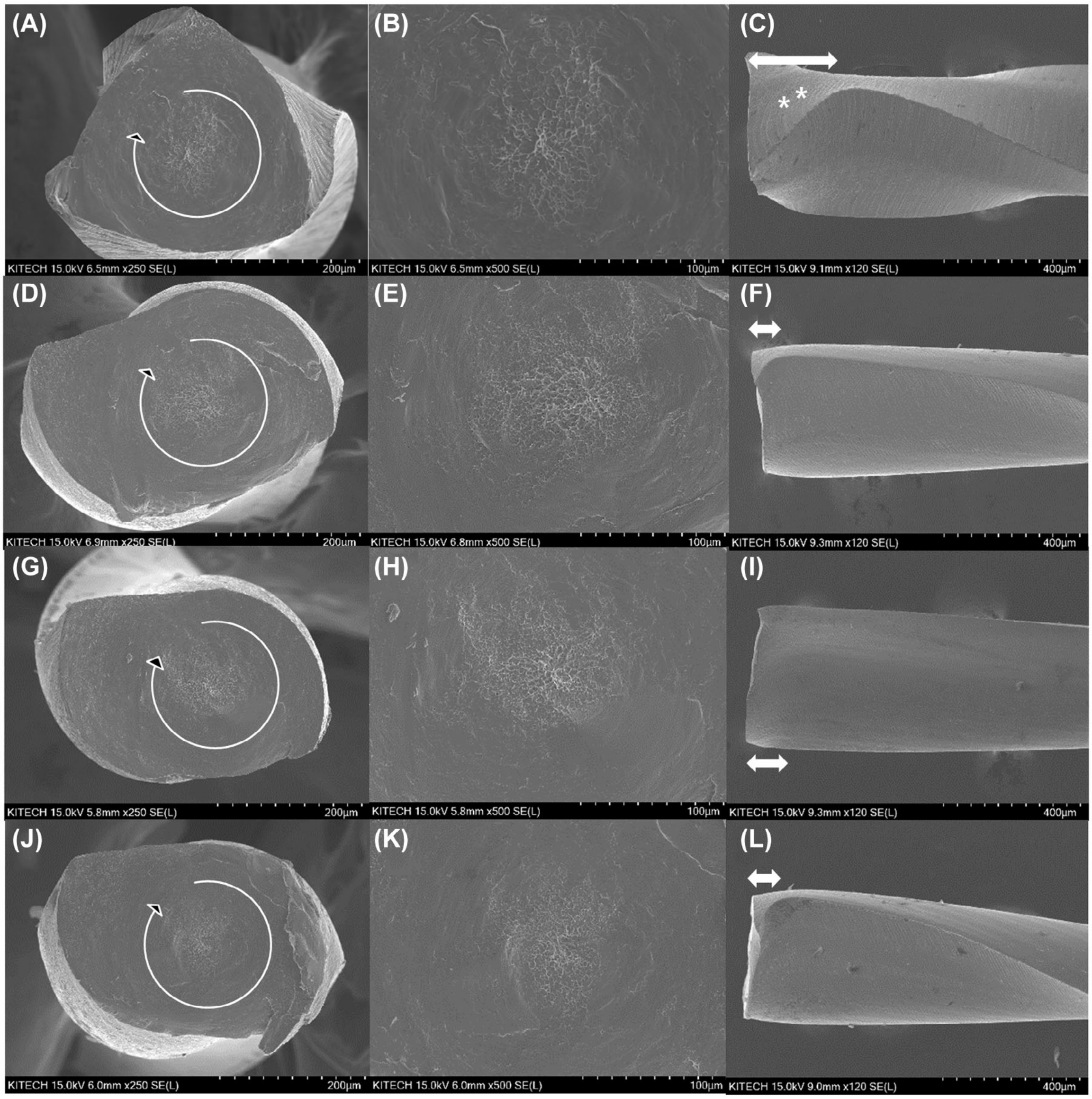

FIGURE 3 Scanning electron microscope images of separated file fragments after the torsional resistance test. ProTaper Gold (PTG): A–C; FQhg: D–F; FQgh: G–I; FQnh: J–L. Cross-sectional views (A, D, G, J) at ×250, enlarged cross-sectional views (B, E, H, K) at ×500, and lateral views (C, F, I, L) at ×120. In the cross-sectional views (A, D, G, J), circular arrows indicate concentric abrasion marks, a typical feature of torsional failure. Dimples are observed in the enlarged cross-sections (B, E, H, K). In the lateral views (C, F, I, L), double arrows denote distortion at the base of the fractured area, and asterisks indicate curved deformation of linear machining grooves.

and chemical composition, but their physical properties may vary due to differences in heat treatment technique and cross-sectional design. The ProTaper file, included as a reference because of its global use, was compared with the modified FQ file to validate the purpose of our experiment. The influence of the manufacturing sequence on file performance was isolated by controlling all other variables.

Although FQnh, which was not heat-treated, showed the lowest cyclic fatigue resistance, the difference from PTG was not statistically significant. The low A*f* temperature of FQnh (24.67˚C) suggests a predominantly austenitic phase at body temperature, which may have limited its flexibility and fatigue resistance. PTG, despite having the highest A*f* (53.13˚C) and a more martensitic structure, exhibited significantly lower fatigue resistance than FQhg and FQgh. This result may be explained by its larger cross-sectional area and higher bending stiffness [21], which could have increased stress concentration during rotation in curved canals, reducing the metallurgical benefits provided by heat treatment. The high resistance to cyclic fatigue observed in FQhg may be attributed to its manufacturing sequence, where heat treatment preceded the grinding process. After heat treatment, machining of NiTi alloys can introduce compressive residual stress on the surface due to non-uniform phase transformation,

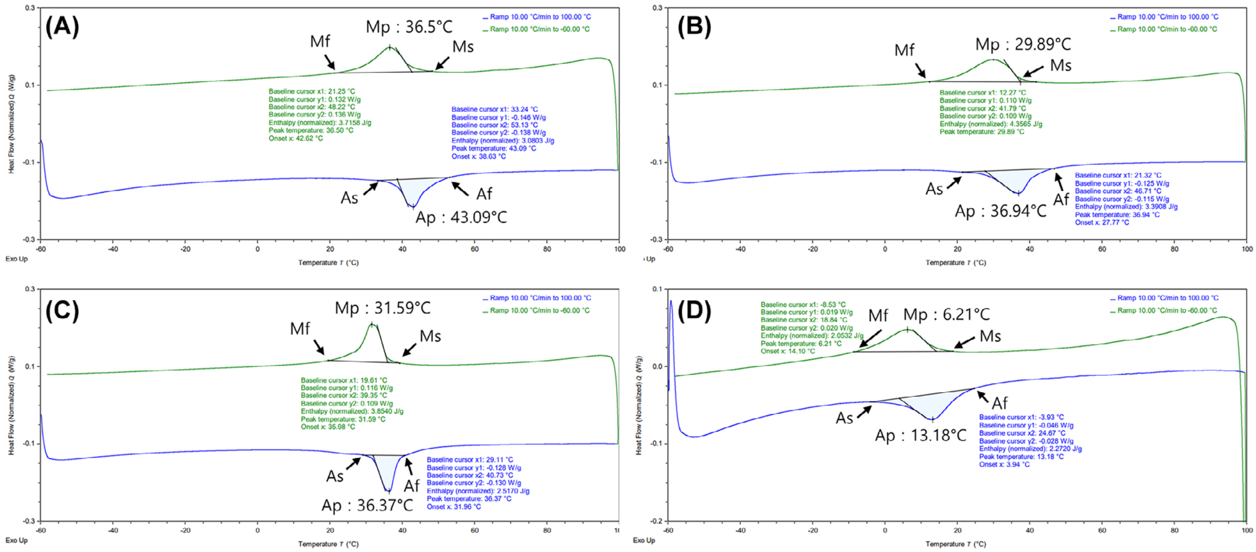

FIGURE 4 Differential scanning calorimetry (DSC) curves of (A) ProTaper Gold, (B) FQhg, (C) FQgh, (D) FQnh (A*s*: austenite start temperature; A*p*: austenite peak temperature; A*f*: austenite finish temperature; M*s*: martensite start temperature; M*p*: martensite peak temperature; M*f*: martensite finish temperature).

the material's intrinsic shape memory effect, and localized plastic deformation and thermal gradients during processing. This compressive stress is believed to enhance resistance to tensile forces generated during rotation, thereby improving fatigue fracture resistance [22, 23]. A possible hypothesis is that, in the FQhg file, compressive residual stress may have been introduced during the grinding process that followed heat treatment. Such compressive stress is known to inhibit crack initiation and propagation, thereby increasing fatigue resistance. Conversely, if grinding is performed before heat treatment, the beneficial compressive stresses may be relieved during the subsequent heat treatment process, potentially reducing the material's resistance to cyclic fatigue.

Based on the torsional resistance test results, PTG showed significantly higher ultimate strength than all FQ groups, while FQhg, FQgh, and FQnh exhibited significantly greater distortion angles compared to PTG. However, no statistically significant difference in toughness was found among the groups. These findings suggest that PTG, due to its larger cross-sectional area and increased structural rigidity, can endure greater torsional loads before fracturing [24]. This structural rigidity contributes to its superior torsional strength, but also limits its capacity for angular deformation, resulting in a lower distortion angle. In contrast, the FQ files demonstrated greater flexibility, as evidenced by their higher distortion angles. The flexibility of the FQ files appears to result from the combined effect of a higher martensite content induced by heat treatment, a double-S cross-sectional design [25]. These factors collectively enhance the file's ability to deform plastically and resist cyclic fatigue. However, the increased flexibility observed in the FQ groups was associated with reduced torsional strength, as evidenced by their lower maximum torque values compared to PTG. Despite these differences, the torsional toughness, which represents the total energy absorbed until fracture, did not show a statistically significant difference among the groups. This suggests that although the modes of energy absorption differed, the total amount of energy required to induce fracture was similar across the groups.

PTG exhibited the highest bending stiffness among all groups. FQhg and FQgh showed the lowest values, with no statistically significant difference between them. These findings indicate that PTG has a considerably more rigid structure, which may contribute to its limited flexibility when navigating curved canals. The high stiffness observed in PTG can be attributed to its larger cross-sectional area and triangular design, both of which increase resistance to flexural deformation. In contrast, FQhg and FQgh, which were manufactured with heat treatment either before or after the grinding process, demonstrated significantly lower bending stiffness. This suggests that the increased martensitic content resulting from heat treatment contributed to greater flexibility [26]. In addition, the reduced cross-sectional area and smaller taper of the FQ files likely result in lower rigidity at the tip compared to PTG, further enhancing their ability to adapt to canal curvature. FQnh, which was not heat-treated, showed intermediate stiffness values, higher than FQhg and FQgh, but lower than PTG, likely to reflect its superelastic phase dominance and less thermally modified structure. These results indicate that heat treatment enhances the flexibility of NiTi instruments by reducing bending stiffness, potentially improving canal negotiation and minimizing the risk of transportation in curved canals [27].

DSC demonstrated that the FQgh NiTi file, processed via grinding before heat treatment, exhibited a narrower and more intense austenite peak compared to FQhg, which underwent heat treatment prior to grinding. In FQhg, subsequent grinding likely introduced residual compressive stresses and localized plastic deformation, resulting in a more heterogeneous microstructure and a broader, less defined phase transformation range. Conversely, because FQgh's thermal treatment followed mechanical processing, residual stresses were more effectively relieved, leading to a more homogeneous structure and sharper, synchronized austenite transformation behavior. Despite FQhg displaying a broader and less pronounced transformation peak in DSC, the compressive residual stress introduced by grinding (prior to heat treatment) likely enhances cyclic fatigue resistance by hindering crack initiation and growth under repeated loading. FQnh, which received no heat treatment, showed lower transformation temperatures (A*f*: 24.67°C, A*p*: 13.18°C). The predominance of the austenitic phase at body temperature may be associated with the file's relatively high stiffness and low fatigue resistance [28]. PTG displayed the highest overall transformation range, with an A*f* of 53.13°C and a peak temperature of 43.09°C, indicating that it remains martensitic even above physiological temperatures. Although this may enhance flexibility in theory [29], the higher stiffness and larger cross-sectional design of PTG might counteract these benefits, potentially explaining its lower cyclic fatigue resistance relative to FQhg.

The results demonstrate that both the application and sequence of heat treatment influence the thermal and mechanical properties of NiTi rotary instruments. The delayed transformation seen in FQhg may offer potential advantage in maintaining a flexible martensitic structure during shaping procedure. Although some differences were observed between FQhg and FQgh depending on the manufacturing sequence, the extent of variation was relatively minor. These findings suggest that the sequence of grinding and heat treatment may influence certain properties, such as cyclic fatigue resistance and torsional strength. However, the overall effect was limited, and the null hypothesis regarding the manufacturing sequence can be partially accepted.

A primary limitation of this study is that torsional and cyclic loading were evaluated independently, although both types of stress are applied simultaneously during clinical root canal instrumentation. This separation may not fully reflect the actual mechanical environment experienced by NiTi files during use. Further studies are needed to investigate file performance under combined loading conditions that better simulate clinical situations. Another limitation is that the experimental conditions did not fully simulate the clinical environment, including factors such as root canal anatomy, intracanal temperature, and moisture. As a result, the mechanical behavior of NiTi files observed in this study may not entirely reflect their performance during actual clinical procedures. A further limitation is that the specific alloy composition and heat treatment protocols of the tested instruments were not fully disclosed by the manufacturers. This lack of information may have restricted a more detailed interpretation of the observed thermal and mechanical behaviors.

CONCLUSION

Within the limitations of this study, the manufacturing sequence of grinding and heat treatment did not have a substantial impact on the performance of NiTi rotary instruments. Post-heat-treatment grinding improved cyclic fatigue resistance, but effects on other properties were minor, with limited clinical implications. Therefore, while the processing sequence may allow minor optimizations (particularly in cyclic fatigue resistance), core factors such as cross-sectional design and heat treatment protocol remain the primary determinants of NiTi instrument performance, with greater relevance for both manufacturers and clinicians.

AUTHOR CONTRIBUTIONS

Conceptualization: Bu N, Kim HC, Kwak SW. **Formal analysis**: Bu N, Ha JH. **Investigation**: Bu N, Kwak SW. **Methodology**: Ha JH, Kim HC, Kwak SW. **Writing—original draft**: Bu N, Kwak SW. **Writing—review and editing**: Ha JH, Kim HC, Kwak SW.

CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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