BASIC RESEARCH – TECHNOLOGY

Effect of Heat Treatment on Mechanical Properties of Nickel-Titanium Instruments

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ABSTRACT

Introduction: The aim of this study was to evaluate the torsional resistance, cyclic fatigue resistance, and bending stiffness of nickel-titanium (NiTi) file systems with different heat treatments and cross-sectional designs. **Methods:** WaveOne Primary treated with memory-triple (MT) heat treatment (WOMT) was compared with WaveOne Primary (WO) and WaveOne Gold Primary (WOG). Torsional resistance test was performed using a customized device, and the distortion angle, ultimate strength, and toughness were evaluated. For cyclic fatigue resistance test, the instruments were reciprocated with continuous 4 mm up-and-down movement until fracture in a customized device, and the time to fracture was compared. Fracture surfaces of each group were examined under the scanning electron microscope. Bending stiffness was measured using a custom-made device. The results were analyzed using one-way analysis of variance and the Tukey's post hoc comparison at a significance level of 95%. **Results:** WOMT showed higher ultimate strength and toughness than the other systems ($P < .05$). WOMT also showed highest cyclic fatigue resistance among the tested groups ($P < .05$). WO had the highest bending stiffness than others, whereas WOMT had a larger residual angle than others ($P < .05$). **Conclusions:** This new MT heat treatment technique makes NiTi file more flexible and improves its mechanical properties. In addition, the effect of heat treatment on flexibility was found to be more significant than that of the cross-sectional area. *(J Endod 2023;■:1–7.)*

KEY WORDS

bending stiffness; cyclic fatigue resistance; heat treatment; memory-triple; nickel-titanium file; torsional resistance

SIGNIFICANCE

The recently introduced memory-triple (MT) heat treatment technique has proven to be advantageous in enhancing the fracture resistance of nickel-titanium files.

The primary objective of root canal treatment is to achieve disinfection of the pulp and the entire root canal system and to prevent the development of periradicular periodontitis[1,2]. The procedures involving the shaping and cleansing of the root canal system play a crucial role in addressing microbial challenges therein[3,4]. Nickel-titanium (NiTi) instruments are commonly used for root canal treatment due to capacity to yield superior outcomes compared with stainless steel hand instruments in terms of canal shaping efficiency and quality of canal shaping with fewer procedural errors[5,6]. Nonetheless, when a root canal is severely calcified or curved, there is a risk of instrument separation[7,8].

Fracture of NiTi instruments is attributed to either cyclic fatigue or torsional failure[7,8]. Cyclic fatigue fracture ensues as a consequence of repetitive compressive and tensile stresses imposed on the outer portion of a file during rotational movement within a curved root canal. In contrast, torsional fracture transpires when the file's tip becomes firmly entrapped within a narrow and calcified root canal, causing the file's shank to persist in its rotational motion. The success rate of removing a separated instrument varies from 33% to 95%[9,10]. The variation in success rates is due to the several factors, such as the position of the fractured instrument in the canal, type of material, instrument size, and canal anatomy[9,11]. The retrieving of the fractured instruments is very difficult and time-consuming. Intracanal separation of instruments may impede the efficacy of cleaning and shaping procedures within the root canal system[12].

To achieve safe and effective root canal preparation, recent advancements in metallurgical techniques and innovative designs have been incorporated to improve fracture resistance of NiTi files. The application of heat treatment during either the NiTi production process or subsequent to file manufacturing can induce alterations in the phase transformation temperature[13,14]. Instruments exhibiting a higher martensitic phase content tend to display increased flexibility owing to their inherent capacity for phase reorientation within the twinned structure[15]. In comparison with their austenitic

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counterparts, martensitic instruments exhibit superior resistance to cyclic fatigue[14,15]. Another factor that could affect fracture resistance is the cross-sectional design. Previous studies have elucidated that a reduction in the core area contributes to enhance the fatigue resistance of the instrument[16,17]. The geometrical characteristics of instrument cross-sections also influence both fracture strength and contact area between the instrument and root canal wall[16].

WaveOne Gold (Dentsply Sirona, Ballaigues, Switzerland) and Reciproc Blue (VDW, Munich, Germany) were introduced to the market with optimized mechanical properties through thermal treatment of the NiTi alloy. The Gold heat treatment for WaveOne Gold, distinct from the M-wire, involves a slow heating and cooling process, which increases the flexibility of the files[18]. In contrast, Reciproc Blue incorporates a blue heat treatment, resulting in the formation of a thin blue titanium oxide layer on the surface, consequently elevating both flexibility and fatigue resistance[19,20]. More recently, an innovative heat treatment approach known as "memory-triple (MT) heat treatment" has been used in the development of EndoRoad files (Maruchi, Wonju, Korea). The manufacturer of this new file system asserts that this specialized heat treatment induces the creation of the R-phase within the range of the body temperature. This leads to a symmetric presence of R-phase, yielding a substantial enhancement in flexibility and cyclic fatigue resistance of the files when compared with previous heat treatment methodologies.

No studies investigating the impact of this new MT heat treatment were found. Moreover, the relationship between cross-sectional design and heat treatment on the physical properties of NiTi instruments is not well established. Therefore, the purpose of this study was to evaluate the torsional resistance, cyclic fatigue resistance, and bending stiffness of NiTi file systems with different heat treatments and cross-sectional designs. The null hypothesis is that heat treatment and cross-sectional area do not have an influence on the mechanical properties of NiTi instruments.

MATERIALS AND METHODS

WaveOne Primary with MT heat treatment (WOMT; Dentsply Sirona) (#25/.08v) was fabricated for this study. WaveOne Primary (WO) (#25/.08v) was chosen as a control group. To evaluate the physical properties based on heat treatment and cross-sectional differences, WOMT and WaveOne Gold Primary (WOG) (#25/.07v) were compared.

G*Power Version 3.1.9.7 (Heine Heinrich Universität, Düsseldorf, Germany) was used for sample size calculation with an effect size of 0.75, α error of 0.05, and power of 0.95 in accordance with a previous study[21]. The indicated sample size was 33 samples for each test, and 12 new files were included in each of the 3 groups.

The evaluation of torsional resistance was carried out in accordance with the guidelines stipulated in ISO 3630-1, using a specially designed apparatus (AEndoS; DMJ System, Busan, Korea). The 3 mm of file tip was securely clamped between brass plates and subjected to counterclockwise rotation at a speed of 2 rpm until fracture occurred (n = 12 per group). The maximum torsional load and distortion angle were recorded using a torque sensor. The determination of toughness was achieved by computing the values of ultimate strength and distortion angle.

A customized device (EndoC; DMJ System) was used for conducting a cyclic fatigue resistance test. An artificial canal with a curvature of 45° and a radius of 5 mm was made using tempered steel to simulate clinical conditions. The instruments from WO, WOG, and WOMT groups were subjected to a reciprocating motion with a continuous 4-mm up-and-down movement using the “WaveOne All” program, as recommended by the manufacturer, until fracture occurred (n = 12 per group). The moment of instrument fracture was visually and audibly detected, and the time to fracture was recorded. The length of the fractured file tip was measured using a digital microcaliper (Mitutoyo, Kawasaki, Japan) at × 10 magnification under a dental operating microscope (Zeiss Pico; Carl Zeiss MediTex, Dublin, CA).

Bending stiffness was assessed using a custom-made device (AEndoS). The tip of the instrument was fixed 3 mm from the tip and bent at a 45° angle relative to the long axis at a speed of 2 rpm (n = 12 per group). Bending stiffness was recorded, and the residual length between the bent file and the initial position was measured using a digital microcaliper. The value “a” remained fixed, while the value “b” was measured using the digital microcaliper. The residual angle (θ) was calculated as “Arctan (b/a)” [$\theta = \tan^{-1}$(b/a), residual angle].

The fractured fragments from the experimental group were examined using a scanning electron microscope (SEM). Five fractured fragments were selected from each group to determine the topographic features of the fractured instruments at various magnifications.

Differential scanning calorimetry (DSC) analysis was used to determine the phase transformation temperature values. DSC analysis was conducted on 3 NiTi instruments for each experimental group, and the phase transition temperature of each file was measured.

The normality of the distributions was verified using a Shapiro-Wilk test, and the results indicated that the data followed a normal distribution. For instrument comparisons, one-way analysis of variance was performed, followed by Tukey’s post hoc comparison test. All of these statistical analyses were carried out using SPSS software for Windows version 11.0 (SPSS, Chicago, IL) with a significance level set at 95%.

RESULTS

The mean mechanical properties of WO, WOG, and WOMT under torsional resistance, cyclic fatigue, and bending resistance test are presented in Table 1. WOMT exhibited a significantly higher ultimate strength compared with WO and WOG ($P < .05$). WO showed a smaller distortion angle, while WOG and WOMT had comparable results ($P > .05$). WOMT showed the highest toughness, followed by WOG and WO, with no significant difference between WO and WOG ($P > .05$). WOMT showed the longest time to cyclic fatigue failure, followed by WOG and WO ($P < .05$). The residual angle of WOMT was significantly greater than that of WOG and WO ($P < .05$). WO had the highest bending stiffness among the tested groups, whereas WOG and WOMT had comparable results ($P > .05$).

The SEM images of the separated fragment surface showed typical features of torsion fracture, which include the presence of a crack initiation origin accompanied by concentric abrasion marks (Fig. 1*A, E,* and *I*). After the cyclic fatigue test, typical patterns were observed, including 1 or more crack initiation origins and an overload fast fracture zone (Fig. 2*A, E,* and *I*). After the torsional test, distortions in lateral aspect of WO, WOG, and WOMT groups were observed, whereas the lateral aspects of each file system after cyclic loading did not show any specific features (Fig. 1*C, G*, and *K*, Fig. 2*C, G*, and *K*).

Figure 3 presents the DSC curves for each NiTi instrument. The DSC curves for WO and WOG showed a single peak on the heating curves (Fig. 3*A* and *B*), whereas the DSC curve for WOMT before experiments displayed double peaks (W-shaped peaks) on the heating curve (Fig. 3*C*). The austenite starting

TABLE 1 - Mean Mechanical Properties of WaveOne, WaveOne Gold, and WaveOne MT Under Torsional Resistance, Cyclic Fatigue Resistance, and Bending Stiffness Test (Mean ± Standard Deviation)

	Torsional resistance			Cyclic fatigue resistance		Bending stiffness	
System	Ultimate strength (N·cm)	Distortion angle (°)	Toughness (°N·cm)	Time to fracture (second)	Fracture fragment length (mm)	Residual angle (°)	Bending stiffness (N·cm)
WaveOne	1.28 ± 0.10[b]	322.67 ± 29.98[b]	278.56 ± 23.47[b]	161.83 ± 28.30[c]	41.17 ± 9.12[a]	8.81 ± 0.66[c]	1.52 ± 0.25[a]
WaveOne Gold	1.16 ± 0.08[c]	422.86 ± 46.11[a]	306.70 ± 49.00[b]	217.83 ± 22.82[b]	22.58 ± 1.24[b]	10.65 ± 0.51[b]	0.86 ± 0.14[b]
WaveOne MT	1.46 ± 0.11[a]	466.78 ± 53.54[a]	438.55 ± 54.52[a]	549.75 ± 41.78[a]	36.83 ± 7.88[a]	33.37 ± 0.83[a]	0.81 ± 0.37[b]

[abc]Different superscript letters indicate statistical differences between groups in vertical columns ($P < .05$).

temperatures of WO, WOG, and WOMT were around 29°C, 37°C, and 53°C, respectively. These transition temperatures indicate the phases in which each NiTi instrument exists at room temperature (25°C). Therefore, the WO and WOG groups primarily exhibited a martensitic phase at room temperature and a combination of martensitic and austenitic phases at body temperature (37°C). In contrast, the WOMT group remained in the martensitic phase at both room and body temperatures.

DISCUSSION

The geometric designs, metallurgical properties, and thermomechanical treatments of the NiTi file systems have an effect on their mechanical behaviors[15,22,23]. Despite a comprehensive literature review, there has been no study examining the mechanical properties of the new MT heat treatment on NiTi files. In the present study, to evaluate the effect of heat treatment and cross-sectional design on mechanical properties, 3 experimental groups were tested, WO, WOG, and WO, with the new MT heat treatment.

WO is made of M-wire and features a convex triangular cross-section. On the other hand, WOG, a next generation instrument

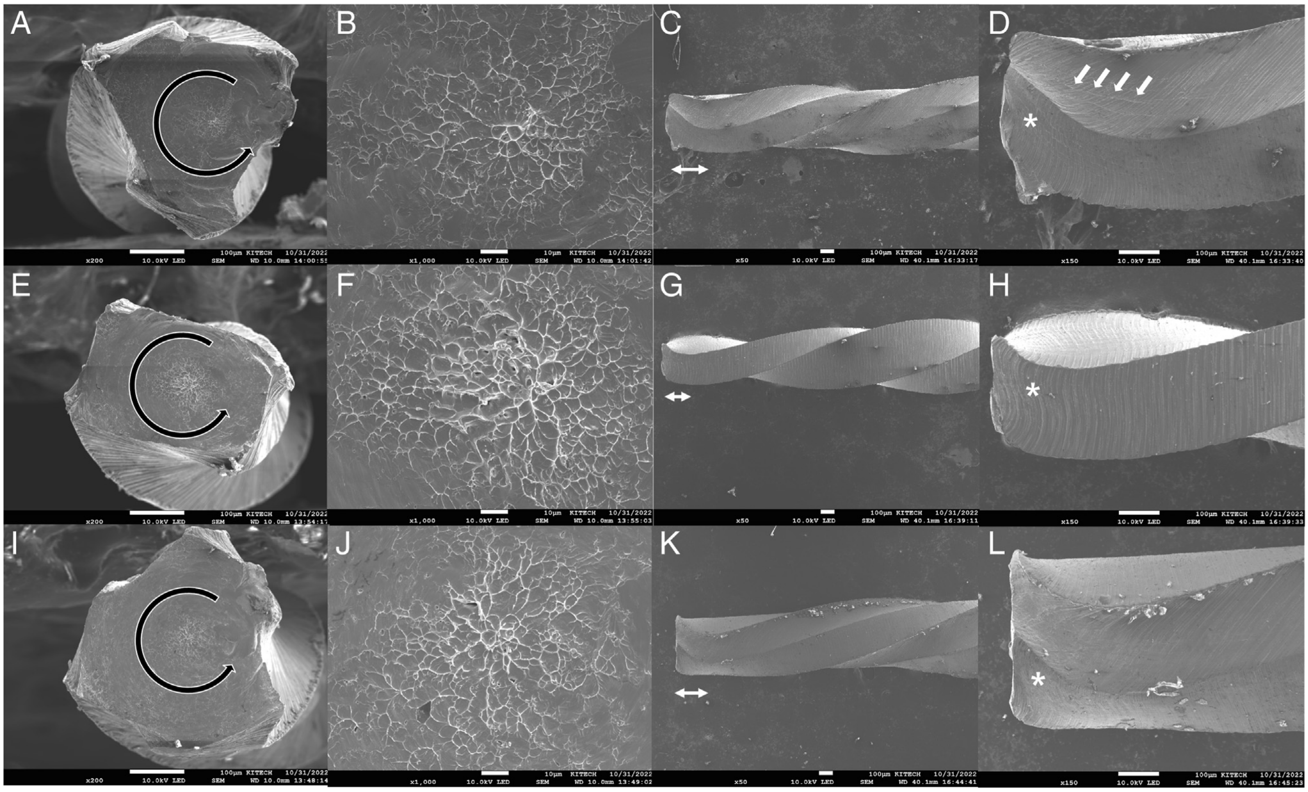

FIGURE 1 – Scanning electron microscope images of separated fragments (WaveOne: *A, B, C, D*; WaveOne Gold: *E, F, G, H*; WaveOne MT: *I, J, K, L*) after torsional resistance test. Cross-sectional aspects of fractured surface (*A, E, I*) showed typical features of torsional fractures with circular arrow indicating the concentric abrasion mark. Skewed dimples near the center of rotation were observed (*B, F, J*: at ×1000 magnification). The longitudinal view (*C, G, K*) showed the distortion near the fractured surface (double arrows). The magnified lateral aspects from the specimens (*D, H,* and *L*) show a curved deformation of the straight machining groove is observed near the fractured surface (*). The WaveOne (*D*) showed the longitudinal microcracks (open white arrows).

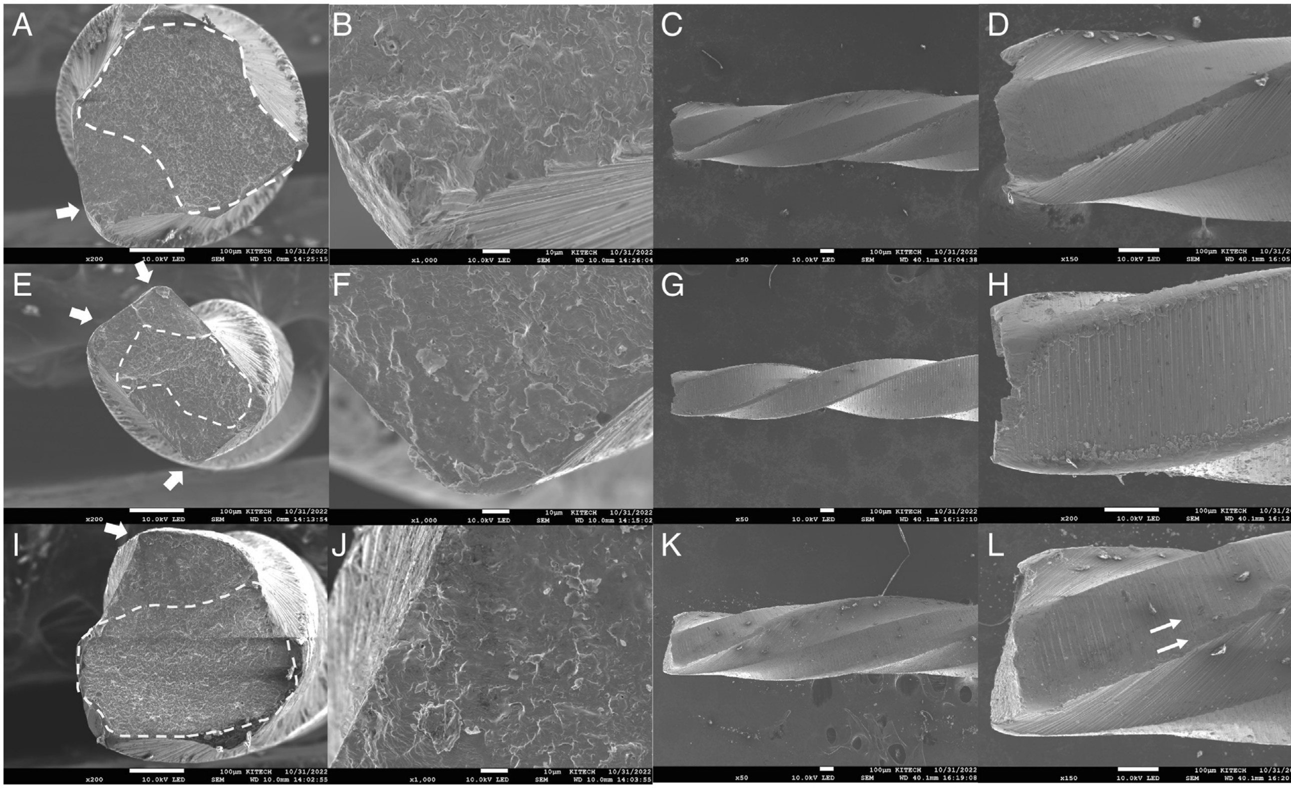

FIGURE 2 – Scanning electron microscope images of separated fragments (WaveOne: *A, B, C, D*; WaveOne Gold: *E, F, G, H*; WaveOne MT: *I, J, K, L*) after cyclic fatigue test. In cross-sectional view of fractured surface (*A, E, I*), the white arrows indicate the crack initiation origin, and the dotted lines indicate the overload fast fracture zone (*B, F, J*: at ×1000 magnification). In the magnified lateral view (*C, G, K*), the white arrows indicate microcracks near the fractured area (*D, L*: at ×150 magnification, *H*: ×200 magnification).

following WO, is made of gold wire and has a parallelogram structure with 2 cutting edges. A new file called WOMT was fabricated to examine the effect of cross-sectional area and heat treatment on mechanical properties. WOMT shares the same cross design as WO, but this file system is manufactured using the new MT heat treatment. The variations in cross-sectional geometries contribute to distinct mechanical characteristics among different types of instruments. As a result, assessing the exclusive impact stemming solely from the heat treatment of the NiTi alloy posed certain limitations. To address this, comparing the physical properties of NiTi file systems, which share the same cross-sections and tapers but undergo different heat treatments, emerges as a viable approach to gain an understanding of the influence exerted by heat treatment on NiTi files[24]. In this study, the same size of files (ISO #25) was tested, as it is a size commonly used during instrumentation.

According to the results of the present study, WOMT demonstrated significantly higher resistance to torsion and cyclic fatigue compared with both WO and WOG files. Furthermore, WOMT exhibited the lowest bending stiffness among the tested groups, followed by WOG and WO. Thus, the null hypothesis was rejected. These findings are consistent with previous studies that compared the 3 parameters mentioned previously between M-wire and CM wire[25,26].

From the torsional resistance test, WOMT had a significantly higher distortion angle, ultimate strength, and toughness compared with WO and WOG. This result indicating that the ultimate strength of WOMT is higher than that of WO contradicts previous studies suggesting that torsional fracture resistance is generally reduced by heat

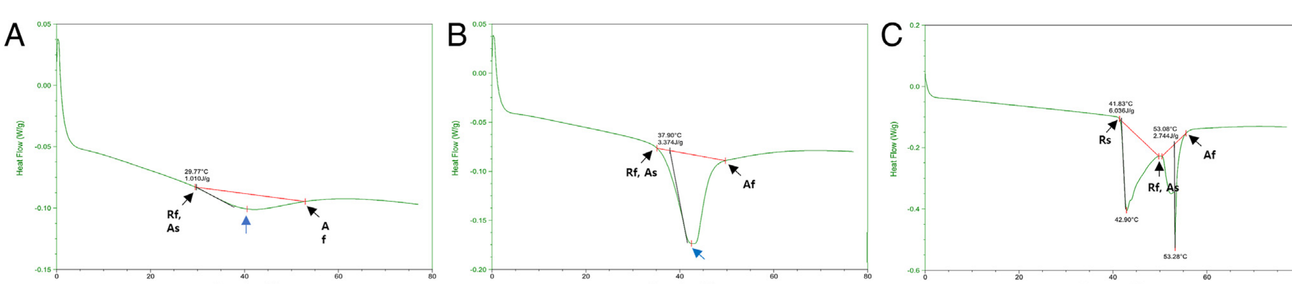

FIGURE 3 – Representative heating curves from differential scanning calorimetry of (*A*) WaveOne Primary, (*B*) WaveOne Gold Primary, (*C*) WaveOne MT Primary. Af, austenite finish temperature; As, austenite start temperature; MT, memory-triple; Rs, R-phase start temperature; Rf, R-phase finish temperature.

treatment[27,28]. This contrasting finding may be attributed to the high flexibility of WOMT, which allows for greater deformation capacity and results in higher torsional strength values. When comparing WOMT and WOG, WOMT showed a higher ultimate strength despite having a larger cross-sectional area than WOG[29]. This suggests that the impact of heat treatment on torsional resistance may be more significant than the cross-sectional area.

In the cyclic fatigue resistance test, WOMT exhibited the highest cyclic fatigue resistance among all the tested groups. This finding aligns with a previous study[30], which reported that WOG has greater cyclic fatigue resistance compared with WO. Kaval et al[31] reported that cyclic fatigue could be influenced by cross-sectional area, indicating that instruments with smaller cross-sections tend to have better resistance. However, in the present study, WOMT, despite having a larger cross-sectional area and taper than WOG, displayed superior resistance to cyclic fatigue. This result may be attributed to the MT heat treatment applied to WOMT, suggesting that the impact of heat treatment on cyclic fatigue resistance could be more significant than the effect of cross-sectional area.

The result of this study showed that WOMT and WOG exhibited lower bending stiffness than WO. WO made of M-wire would have greater bending stiffness than WOG and WOMT. When comparing WOMT and WOG, WOMT with a larger cross-sectional area and taper showed a larger residual angle than WOG. This finding indicates that the MT instrument has more flexible properties at room temperature. From the DSC analysis, it can be considered that MT heat treatment imparts these flexible properties. Therefore, MT heat-treated instruments have less resistance to bending motion, allowing them to easily bend in curved canals and shape root canals with less canal transportation tendency.

Regarding thermomechanical treatment, instruments that underwent R-phase heat treatment technology showed improved cyclic fatigue resistance[32]. According to the manufacturer, the novel heat treatment process applied in the production of the WOMT files enables the generation of R-phase within the body temperature range, resulting in a symmetric R-phase that improves the flexibility and cyclic fatigue resistance of these files. In the DSC analysis, it was observed that WO and WOG displayed a single peak, while WOMT exhibited double peaks. The first peak of WOMT corresponds to the initial transformation from the martensite phase to the R-phase, and the second peak corresponds to the transformation from the R-phase to the austenite phase (Fig. 3*C*). The austenite starting temperature of WOMT was above 53°C and it can be assumed that WOMT contains the martensite phase in the temperature of room and body temperature. Because of the increased amount of the martensite phase, martensite dominant instruments such as WOMT are more flexible with an enhanced resistance to cyclic fatigue and exhibit a greater angle of rotation. As the energy that can be absorbed by MT heat treatment increases, torsional and cyclic fatigue resistance increases.

The cross-sectional design of the instrument affects the mechanical properties of NiTi files. Baek et al[33] evaluated the effect of geometric design on mechanical properties of NiTi instruments and reported that, for the same alloy, an increased cross-sectional area resulted in higher torsional fracture resistance but lower cyclic fatigue resistance. Kim et al[34] also reported that a larger cross-sectional area would lead to higher torsional stiffness but lower cyclic fatigue resistance. In this study, however, it was observed that WOMT with a larger cross-sectional area demonstrated higher cyclic and torsional resistance compared with WOG. Therefore, it is reasonable to conclude that the flexibility or mechanical properties of the instruments are more influenced by the heat treatment rather than the cross-sectional design.

This *in vitro* study, comparing the physical properties of the file with and without heat treatment in the same file, is expected to yield similar results for rotary files. Expanding the investigation to include various NiTi files, heat treatment methods, kinetics, and cross-sectional designs would offer a more comprehensive understanding of how these factors interact and influence the performance of NiTi instruments. In addition, it is essential to consider that the properties of NiTi files observed in the laboratory setting may differ from those encountered in clinical situations. Therefore, setting the experimental conditions that better simulate clinical scenarios is necessary for more relevant research.

In conclusion, within the limitations of this study, it is possible to generalize that MT heat treatment technique could make higher torsional and cyclic fatigue resistance compared with M-wire and Gold wire instruments. In addition, the impact of heat treatment on flexibility was found to be more significant than that of the cross-sectional area. The enhanced flexibility observed in MT wire instruments can be advantageous in improving the quality of root canal shaping during clinical practice, surpassing the performance of M-wire and conventional NiTi instruments.

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The authors deny any conflicts of interest related to this study.

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