

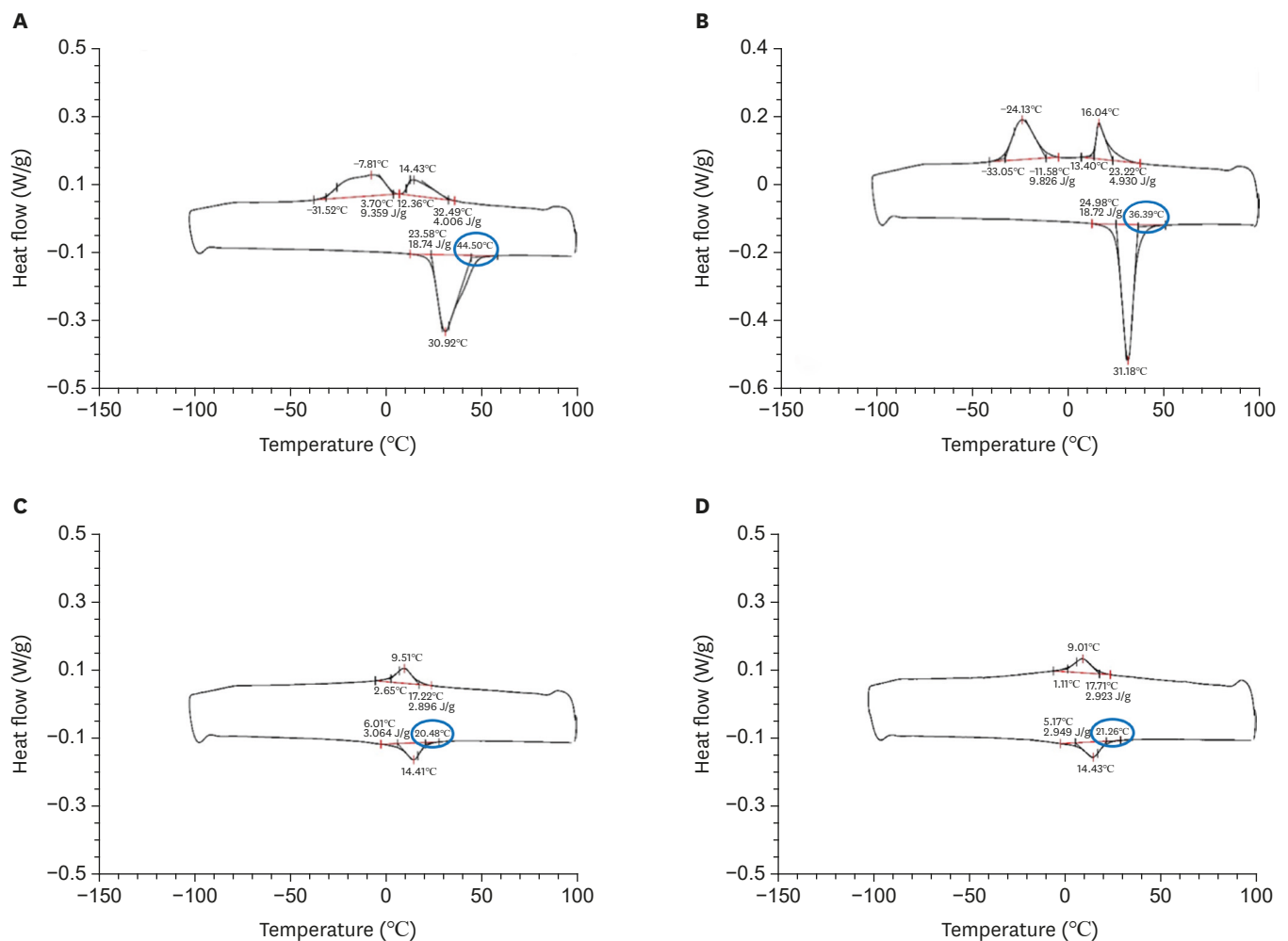
How heat treatments influence NiTi file properties
Table 2. Transformation temperatures (°C) for the nickel-titanium rotary instruments that were tested for 3 samples of each instrument

Instrument	Cooling				Heating			
	R _s	R _f	M _s	M _f	R _s	R _f	A _s	A _f
M3 Pro Gold size 20/0.04	32.49 ± 0.20	12.36 ± 0.22	3.70 ± 0.17	-31.52 ± 0.23	-	-	23.58 ± 0.12	44.50 ± 0.18
M3 Pro Gold size 25/0.04	23.22 ± 0.19	13.40 ± 0.24	-11.58 ± 0.16	-33.05 ± 0.22	-	-	24.98 ± 0.15	36.39 ± 0.17
M3 Rotary size 20/0.04	-	-	17.22 ± 0.16	2.65 ± 0.22	-	-	6.01 ± 0.14	20.48 ± 0.16
M3 Rotary size 25/0.04	-	-	17.71 ± 0.18	1.11 ± 0.21	-	-	5.17 ± 0.12	21.26 ± 0.19

Data were shown with mean ± standard deviations.

M_s, martensite transformation starting point; M_f, martensite transformation finishing point; R_s, R-phase transformation starting point; R_f, R-phase transformation finishing point; A_s, austenite transformation starting point; A_f, austenite transformation finishing point.

clear endothermic peak on the heating curve. On the cooling curve, there were 2 exothermic peaks. The peaks at approximately -8°C and -25°C for the M3 Pro Gold size 20/0.04 and size 25/0.04, respectively, corresponded to the initial transformation from the martensitic phase to the R-phase, and the peaks at approximately 15°C and 20°C for the M3 Pro Gold size 20/0.04 and size 25/0.04, respectively, corresponded to the transformation from the R-phase to the austenitic phase. The DSC diagrams for the M3 Rotary file (**Figure 2C and 2D**) showed a single and defined peak upon cooling and heating. This peak represents the martensitic and reverse transformation between austenite and martensite.


Figure 2. Typical differential scanning calorimetry (DSC) diagram obtained from (A) an M3 Pro Gold size 20/0.04 file, (B) an M3 Pro Gold size 25/0.04 file, (C) an M3 Rotary size 20/0.04 file, and (D) an M3 Rotary size 25/0.04 file. Blue circles indicate the austenite transformation finishing point (A_f) temperatures for each file.

DISCUSSION

Several variables, such as instrument size, taper, cross-sectional design, and manufacturing techniques, affect the clinical performance of endodontic files and their resistance to fracture by torsion and/or cyclic fatigue [18,19].

In this study, cyclic fatigue, maximum torque load, and the angular rotation of the M3 Rotary and new M3 Pro Gold files were evaluated. The M3 Rotary files and M3 Pro Gold files were compared because they have an identical design and geometrical features, but differ only in the heat treatment used in their manufacturing process; thus, the influence of other variables was eliminated in the present study. Moreover, only 2 sizes (sizes 20 and 25 with 0.04 taper) of both instruments were tested because these sizes are commonly used during instrumentation.

According to our results, when the same size of the 2 different instruments was compared, the M3 Pro Gold files exhibited higher cyclic fatigue resistance and angular rotation to fracture than the M3 Rotary files, while no significant difference was observed between the 2 instruments for the maximum torsional strength. These results are probably due to the different NiTi alloys and manufacturing processes [20,21]. In fact, according to the manufacturer, M3 Rotary instruments are made by an advanced memory alloy technology, which increases cyclic fatigue resistance [12]. No data are available on the metallurgical characteristics of this alloy. However, the DSC analysis conducted in this study revealed that the M3 Rotary files seem to be made with an alloy similar to the traditional NiTi. Indeed, M3 Rotary files, as conventional superelastic NiTi [22], have an austenite structure at room and oral temperature, while the M3 Pro Gold files, as CM wire, have a mixture of martensite and austenite at room temperature. In particular, the A_f of the M3 Pro Gold sizes 20 and 25 files were measured to be 44.50°C and 36.39°C, respectively, from their heating curves. This means that at human body temperature (around 37°C) [23], the M3 Pro Gold files are in a mixed austenite and martensite phase. In contrast, the A_f of the sizes 20 and 25 M3 Rotary files were measured to be 20.48°C and 21.26°C, respectively, from their heating curves, which means that M3 Rotary files are in a state of only austenite at human body temperature. Since martensite is less stiff than austenite [24], it is likely that shape memory files present lower stiffness than conventional or M-wire files [25].

Shen *et al.* [11,26] observed increased fatigue resistance in files manufactured via proprietary CM wire processing. The reason for this may be that for a given strain, a more flexible file would experience less stress, allowing for a longer fatigue lifetime [25]. Moreover, there was no significant difference between the M3 Rotary and M3 Pro Gold files in terms of maximum torsional strength. The type of metal alloy, consequently, does not appear to affect torsional strength. These results are in agreement with previous studies [20,21]. However, some previous studies reported that CM wire instruments were associated with a lower torque to failure than conventional or M-wire instruments [25,27]. These differences can be attributed to the different type and size of the tested instruments.

In addition, the angular rotation to fracture of a NiTi file is associated with its ability to be plastically deformed under stress without failing [21]. In this study, the M3 Pro Gold files were significantly more plastic than the M3 Rotary files. In particular, the M3 Pro Gold size 25/0.04 file exhibited the highest angular rotation to fracture. The increase in the plasticity of thermally treated files is thought to be due to the increase of the proportions of the

R-phase and martensite. The R-phase has the lowest shear modulus among the 3 phases [28]. Martensite is more likely to deform than austenite since martensite has a twinning process, which refers to an internal movement of lattices without breaking atomic bonds by absorbing stress [22].

To explain the mechanical behavior observed in both fracture resistance tests, the effects of the size, as well as the cross-section, should not be ignored. The results of the present study show that the size 25 diameter instruments had significantly higher torsional (ultimate) strength than the size 20 diameter instruments. These findings imply that the diameter play an important role in torsional resistance, as previously reported [29,30]. In contrast, size 20 diameter files showed higher cyclic fatigue resistance than size 25 diameter files. These results are in agreement with previous studies reporting that smaller instruments usually showed high flexibility and fatigue resistance [6,10]. In particular, the M3 Pro Gold size 20 file with a 0.04 taper exhibited the highest values of cyclic fatigue resistance. These results are probably due to the benefits of metal treatment associated with the reduced dimensions of the instrument.

Moreover, in a supplementary study, the M3 Rotary size 20 file with a 0.04 taper was found to have a smaller area ($71,665 \mu\text{m}^2$) than the M3 Rotary size 25 file with a 0.04 taper ($80,841 \mu\text{m}^2$), based on a measurement of their cross-sectional area at 5 mm from the tip (D5) under scanning electron microscopy by software (AutoCAD, Autodesk Inc., San Rafael, CA, USA). The same results were found when the cross-sectional area was measured at 5 mm from the tip (D5) of the M3 Pro Gold size 20 file with a 0.04 taper and the M3 Pro Gold size 25 file with a 0.04 taper, considering that M3 instruments with the same tip size and taper had the same cross-sectional area regardless of the heat treatment received (M3 Rotary or Pro Gold). The present findings agree with those of previous studies reporting that a reduced cross-sectional area increased cyclic fatigue resistance but negatively affected torsional resistance [31,32].

The clinical implications of these observations are as follows. A high angle of rotation before fracture in controlled memory files may be beneficial because it may provide clinicians with an indication that plastic/permanent deformation has taken place and that fracture is imminent [25]. In addition, greater flexibility could be useful in clinical situations with high flexural fatigue (*e.g.*, in curved canals).

CONCLUSIONS

Within the limitations of this study, it was observed that the M3 Pro Gold files showed greater flexibility than the M3 Rotary files, with no decrement of other mechanical properties such as torque resistance or angular rotation.

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