

## Research Article



# Cyclic fatigue resistance, torsional resistance, and metallurgical characteristics of M3 Rotary and M3 Pro Gold NiTi files

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### Conflict of Interest

No potential conflict of interest relevant to this article was reported.

### Author Contributions

Conceptualization: Pedullà E, Gambarini G; Data curation: Pedullà E, Gambarini G, Rapisarda E, Chang SW, Testarelli L; Formal analysis: Pedullà E, La Rosa GRM, Rapisarda S, Bruno E; Investigation: Rapisarda E, Testarelli L, Miccoli G; Methodology: Pedullà E, La Rosa G, Lo Savio F; Project administration:

## ABSTRACT

**Objectives:** To evaluate the mechanical properties and metallurgical characteristics of the M3 Rotary and M3 Pro Gold files (United Dental).

**Materials and Methods:** One hundred and sixty new M3 Rotary and M3 Pro Gold files (sizes 20/0.04 and 25/0.04) were used. Torque and angle of rotation at failure ( $n = 20$ ) were measured according to ISO 3630-1. Cyclic fatigue resistance was tested by measuring the number of cycles to failure in an artificial stainless steel canal (60° angle of curvature and a 5-mm radius). The metallurgical characteristics were investigated by differential scanning calorimetry. Data were analyzed using analysis of variance and the Student-Newman-Keuls test.

**Results:** Comparing the same size of the 2 different instruments, cyclic fatigue resistance was significantly higher in the M3 Pro Gold files than in the M3 Rotary files ( $p < 0.001$ ). No significant difference was observed between the files in the maximum torque load, while a significantly higher angular rotation to fracture was observed for M3 Pro Gold ( $p < 0.05$ ). In the DSC analysis, the M3 Pro Gold files showed one prominent peak on the heating curve and 2 prominent peaks on the cooling curve. In contrast, the M3 Rotary files showed 1 small peak on the heating curve and 1 small peak on the cooling curve.

**Conclusions:** The M3 Pro Gold files showed greater flexibility and angular rotation than the M3 Rotary files, without decrement of their torque resistance. The superior flexibility of M3 Pro Gold files can be attributed to their martensite phase.











**Keywords:** Cyclic fatigue; Differential scanning calorimeter; Heat-treatment; M3 Pro Gold; M3 Rotary; Torsional resistance

## INTRODUCTION

The introduction of nickel-titanium (NiTi) rotary instruments has been beneficial for endodontic practice [1,2]. Despite the good mechanical properties of NiTi alloys, the possibility of instrument separation is still a major concern during the clinical use of NiTi files [3]. Many variables contribute to file separation, but the 2 main causes are cyclic fatigue

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and torsional stress [4]. Cyclic flexural fatigue of an instrument occurs when the instrument is rotated in a curved canal by repeated compressive and tensile stresses, while torsional failure occurs when the tip of the instrument binds in the canal, but the motor continues to rotate. The elastic limit of the instrument is exceeded, causing plastic deformation and, subsequently, failure [5].

The variables that contribute to file separation include instrument size and cross-sectional area [6,7], design [8], metal surface treatments [1], and the metallurgical characteristics of the NiTi alloy, among others [9]. Therefore, several manufacturers have developed special forms of thermomechanical processing, with the aim of producing a superelastic NiTi alloy that primarily contains a stable martensitic phase under clinical conditions [10]. In recent years, NiTi rotary instruments made from a NiTi controlled memory wire (CM wire, DS Dental, Johnson City, TN, USA) have been introduced. The manufacturer claims that these instruments have superior flexibility and fatigue resistance to conventional NiTi rotary instruments made from superelastic wire [11].

The M3 Rotary file (M3, United Dental, Shanghai, China) is a newly introduced NiTi instrument that was designed to be used in continuous rotation with an inactive tip and a convex triangular cross-section; it consists of 3 instruments with sizes 20, 25, and 30 tip sizes and a 0.04 taper. In addition, opener and glide path files are also provided [12]. According to the producer, it is manufactured using an advanced memory alloy technology that can increase cyclic fatigue resistance. The manufacturer claims that this instrument allows quick and safe preparation of root canals, especially of curved root canals [12]. Recently, M3 Rotary has been upgraded to M3 Pro Gold (United Dental). These files have the same geometrical and design features as the M3 Rotary files, but undergo a different heat treatment. M3 Pro Gold files are manufactured with a CM wire associated with a gold treatment, which allows greater flexibility [13].

To date, no data are available on the mechanical properties of the M3 Pro Gold file, such as torsional stress and cyclic fatigue resistance. Hence, the aim of this work was to investigate the cyclic fatigue, maximum torque load, angular rotation, and metallurgical characteristics of the M3 Pro Gold file and to compare these findings with those of the M3 Rotary file.

## MATERIALS AND METHODS

Sample size estimation was calculated *a priori* with G\*Power 3.1.9.2 software (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany) [14]. The M3 Rotary size 20, 0.04 taper; M3 Rotary size 25, 0.04 taper; M3 Pro Gold size 20, 0.04 taper; and M3 Pro Gold size 25, 0.04 taper were used in this study. All tested files were 25 mm long, with 20 instruments each consumed in cyclic fatigue and torsional resistance tests. Every instrument was inspected for defects or deformities before the experiment under a stereomicroscope (SZR-10, Optika, Bergamo, Italy); none was discarded. The torsional load was applied until fracture to estimate the mean ultimate torsional strength, and the angle of rotation of the instruments was tested using a custom-made device produced following ISO 3630-1 [4].

Each file was clamped at 3 mm from the tip using a chuck connected to a torque-sensing load cell, after which the shaft of the file was fastened into an opposing chuck able to be rotated with a stepper motor. All tested files were rotated in the clockwise direction at a speed of 2

revolutions per minute until file separation. The torque load (N·cm) and angular rotation (°) were monitored continuously using a torsionmeter (Sabri Dental Enterprises, Downers Grove, IL, USA) at room temperature ( $21^{\circ}\text{C} \pm 1^{\circ}\text{C}$ ), and the ultimate torsional strength and angle of rotation at failure were recorded.

The cyclic fatigue tests were performed using a custom-made device that allowed a reproducible simulation of an instrument confined in an artificial curved canal, as described previously [15,16]. The artificial canal was manufactured by reproducing the instrument size and taper, thus providing the instrument with a suitable trajectory with a  $60^{\circ}$  angle of curvature and a 5-mm radius of curvature. The instruments were rotated at a constant speed of 300 rpm using a 6:1 reduction handpiece (Sirona, Bensheim, Germany) powered by a torque-controlled endodontic motor (Silver, VDW, Munich, Germany). To reduce the friction of the file as it contacted the artificial canal walls, a special high-flow synthetic oil designed for the lubrication of mechanical parts (Super Oil, Singer Co. Ltd., Elizabethport, NJ, USA) was applied. All instruments were rotated until fracture occurred. After positioning the instrument into the artificial canal, as soon as rotation started, timing was initiated. Timing was stopped when instrument breakage was observed. For each instrument, the time to fracture in seconds was recorded by the same operator with a chronometer. The number of cycles to failure for each instrument was calculated by multiplying the time (seconds) to failure by the number of rotations or cycles per second. The length of the fractured file tip was measured using a digital microcaliper (Mitutoyo, Kawasaki, Japan). The fracture surfaces of all fragments were examined under a scanning electron microscope (ZEISS Supra 35VP, Oberkochen GmbH, Oberkochen, Germany) to investigate the topographic features of the fractured instruments.

### Differential scanning calorimetry (DSC)

To investigate the thermal properties of the M3 Rotary and M3 Pro Gold files, DSC analysis (TA Instruments, New Castle, DE, USA) was also carried out [17]. For the DSC analysis, small segments of each file were placed in an aluminum pan on a platinum holder inside the measuring chamber of the DSC instrument. An empty pan was included as reference. The chamber was filled with argon gas with high purity to minimize any oxidation. The exothermic or endothermic energy flow was then recorded by raising the temperature to  $100^{\circ}\text{C}$ , lowering it to  $-100^{\circ}\text{C}$ , and then re-raising it to  $100^{\circ}\text{C}$ . Liquid nitrogen was used as a coolant in this experiment. The heating and cooling rate was  $0.17^{\circ}\text{C}/\text{s}$ . The martensite transformation starting point ( $M_s$ ), martensite transformation finishing point ( $M_f$ ), R-phase transformation starting point ( $R_s$ ), R-phase transformation finishing point ( $R_f$ ), austenite transformation starting point ( $A_s$ ), and austenite transformation finishing point ( $A_f$ ) were determined.

### Statistical analysis

The data were first checked using the Kolmogorov-Smirnov test for normality of the distribution and using the Levene test for the homogeneity of variance. Then, the data were statistically evaluated by the analysis of variance test and the Student-Newman-Keuls test for multiple comparisons (Prism 5.0, GraphPad Software, Inc., La Jolla, CA, USA) with the significance level established at 5% ( $p < 0.05$ ).

## RESULTS

The mean and standard deviation values of cyclic fatigue resistance, maximum torque load, and angle of rotation until fracture for each instrument are presented in **Table 1**.

**Table 1.** Cyclic fatigue, torque, and angle of rotation of the instruments tested

Instrument	Cyclic fatigue (NCF)	Torque (N·cm)	Angle of rotation (°)
M3 Rotary 20/0.04	564 ± 2 <sup>a</sup>	0.53 ± 0.02 <sup>a</sup>	295 ± 1 <sup>a</sup>
M3 Pro Gold 20/0.04	961 ± 2 <sup>b</sup>	0.56 ± 0.01 <sup>a</sup>	494 ± 2 <sup>b</sup>
M3 Rotary 25/0.04	452 ± 2 <sup>c</sup>	0.71 ± 0.02 <sup>b</sup>	392 ± 2 <sup>c</sup>
M3 Pro Gold 25/0.04	836 ± 2 <sup>d</sup>	0.75 ± 0.01 <sup>b</sup>	596 ± 1 <sup>d</sup>

Data were shown with means ± standard deviations. Different superscript letters in the same column indicate statistically significant differences among groups ( $p < 0.05$ ).

NCF, number of cycles to failure.

When comparing the same size of the 2 different instruments, a statistically significant difference in terms of cyclic fatigue resistance ( $p < 0.001$ ) was noted. In particular, the M3 Pro Gold files exhibited higher cyclic fatigue resistance than the M3 Rotary files ( $p < 0.001$ ). In addition, when comparing different sizes of the same instrument, tip size 20 and 0.04 taper showed higher cyclic fatigue resistance than tip size 25 and 0.04 taper ( $p < 0.05$ ). Moreover, the M3 Pro Gold size 20/0.04 exhibited the highest values of cyclic fatigue resistance of all tested instruments ( $p < 0.05$ ).

Regarding the maximum torsional strength, no statistically significant difference was observed when the same size of the 2 different instruments was compared, while there was a significant difference between the different sizes of the same instrument ( $p < 0.05$ ). In particular, tip size 25 and 0.04 taper showed a higher maximum torsional strength than tip size 20 and 0.04 taper ( $p < 0.05$ ).

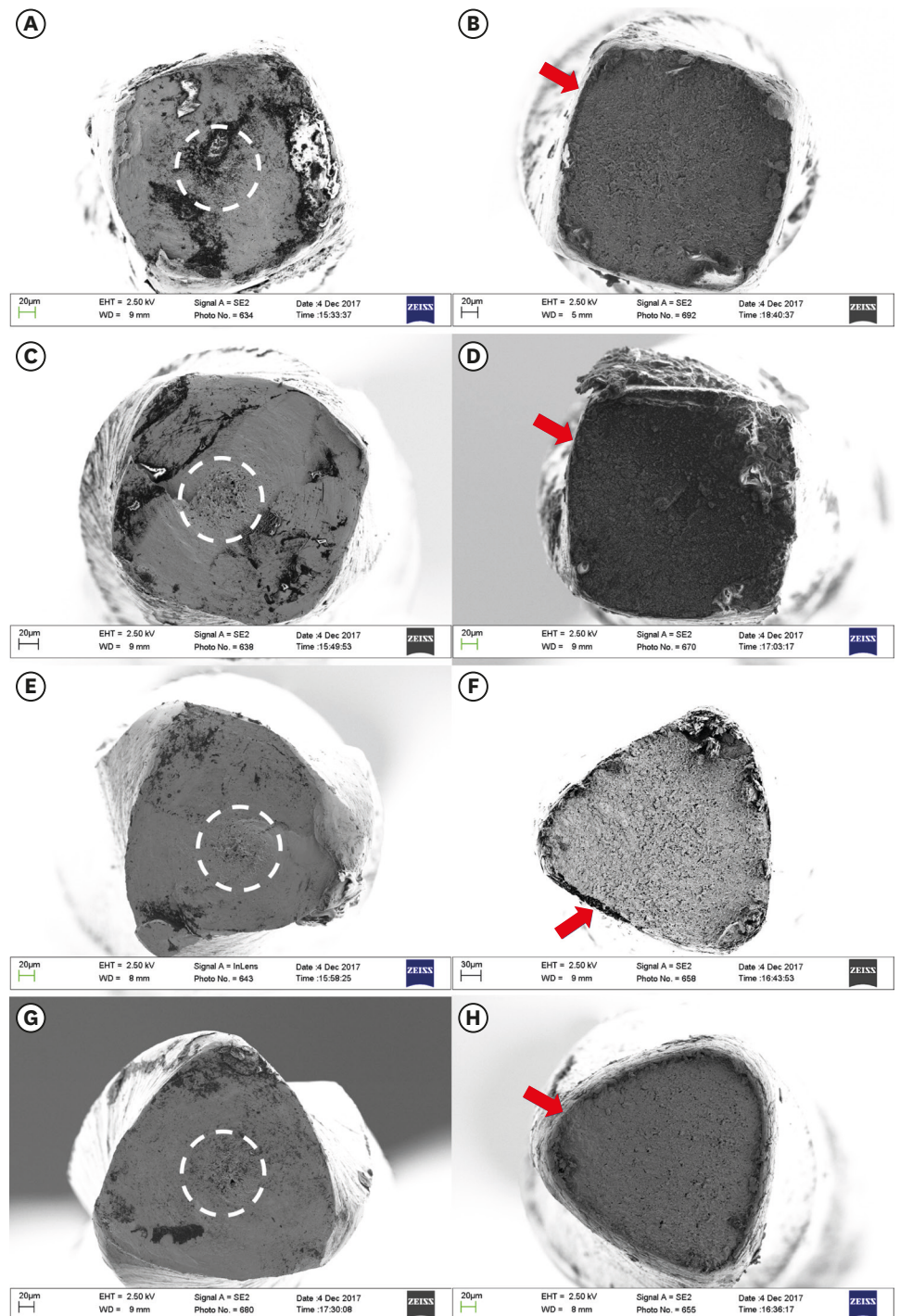
Comparing the same size of the 2 different instruments, the M3 Pro Gold files showed significantly higher angular rotation to fracture (and therefore significantly higher time before torsional fracture) than the M3 Rotary files ( $p < 0.001$ ). In addition, when comparing different sizes of the same instrument, tip size 25 and 0.04 taper was associated with a higher angular rotation to fracture ( $p < 0.05$ ). In particular, M3 Pro Gold size 25/0.04 exhibited the highest angular rotation to fracture of all tested files ( $p < 0.05$ ).

The mean length of the fractured fragment (3 mm and 5.1 mm for instruments tested for torsional and cyclic fatigue, respectively) was not significantly different for any of the instruments tested.

Scanning electron microscopy of the fracture surface showed similar and typical features of cyclic fatigue and torsional failure for all instruments. The crack initiation area and overload fast fracture zone for cyclic fatigue fractures and concentric abrasion marks and the fibrous dimple marks at the center of rotation for torsional failure are shown in **Figure 1**.

The results of DSC analysis ( $A_s$ ,  $A_f$ ,  $R_s$ ,  $R_f$ ,  $M_s$ , and  $M_f$ ) are shown in **Table 2**. M3 Pro Gold showed 1 prominent peak on the heating curve and 2 prominent peaks on the cooling curve. In contrast, the M3 Rotary files showed 1 small peak on the heating curve and 1 small peak on the cooling curve. The peaks observed for the M3 Rotary files were much less intense than those observed for the M3 Pro Gold files, as shown in **Figure 2**.

In particular, **Figure 2A and 2B** shows a typical DSC curve obtained from the M3 Pro Gold files. In the DSC diagram, the exothermic reaction in the upper curve indicates the martensitic transformation in the cooling process, while the endothermic reaction in the lower curve is caused by the reverse transformation from the martensitic phase to the rhombohedral phase (R-phase) and/or austenitic phase in the heating process. There was a



**Figure 1.** Scanning electron micrographs of the fracture surface of the separated fragments. (A, B) M3 Rotary size 20/0.04; (C, D) M3 Pro Gold size 20/0.04; (E, F) M3 Rotary size 25/0.04; (G, H) M3 Pro Gold size 25/0.04. (A, C, E, G) The left column shows the images after the torsional test, with the circular box indicating the concentric abrasion mark; the skewed dimples near the center of rotation are typical features of torsional failure. (B, D, F, H) The right column shows images after the cyclic fatigue test, with red arrows indicating the crack initiation origin; the surface pattern shows dimples and cones observed in the same fracture plane.