Received: 2 August 2022 | Accepted: 9 December 2022
DOI: 10.1111/iej.13880

ORIGINAL ARTICLE

INTERNATIONAL ENDODONTIC JOURNAL | WILEY

Characterization of the file-specific heat-treated ProTaper Ultimate rotary system

Jorge N. R. Martins[1,2,3] | Emmanuel João Nogueira Leal Silva[4,5] | Duarte Marques[1,2,3,6] | Natasha Ajuz[4] | Mário Rito Pereira[1] | Rui Pereira da Costa[1] | Francisco Manuel Braz Fernandes[7] | Marco Aurélio Versiani[8]

[1]Faculdade de Medicina Dentária, Universidade de Lisboa, Lisboa, Portugal
[2]Grupo de Investigação em Bioquimica e Biologia Oral, Unidade de Investigação em Ciências Orais e Biomédicas (UICOB), Faculdade de Medicina Dentária, Universidade de Lisboa, Lisboa, Portugal
[3]Centro de Estudo de Medicina Dentária Baseada na Evidência (CEMDBE) - Cochrane Portugal, Faculdade de Medicina Dentária, Universidade de Lisboa, Lisboa, Portugal
[4]School of Dentistry, Grande Rio University (UNIGRANRIO), Rio de Janeiro, Brazil
[5]Fluminense Federal University, Rio de Janeiro, Brazil
[6]LIBPhys-FCT UID/FIS/04559/2013, Lisboa, Portugal
[7]CENIMAT/I3N, Department of Materials Science, NOVA School of Science and Technology, Universidade NOVA de Lisboa, Caparica, Portugal
[8]Dental Specialty Center, Brazilian Military Police, Belo Horizonte, Brazil

Correspondence
Jorge N. R. Martins, Faculdade de Medicina Dentária da Universidade de Lisboa, Cidade Universitária, Lisboa 1649-003, Portugal.
Email: jnr_martins@yahoo.com.br

Funding information
Fundação para a Ciência e a

Abstract

Aim: To compare design, metallurgy and mechanical performance of the ProTaper (PT) Ultimate system with instruments of similar dimensions from the ProGlider, PT Gold and PT Universal systems.

Methodology: New PT Ultimate instruments ($n = 248$) were compared with instruments of similar dimensions from ProGlider ($n = 31$), PT Gold ($n = 155$) and PT Universal ($n = 155$) systems regarding their number of spirals, helical angle, blade symmetry, tip geometry, surface finishing, nickel/titanium ratio, phase transformation temperatures and mechanical performance. One-way ANOVA and nonparametric Mood's median tests were used for statistical comparison ($\alpha = 5\%$).

Results: All instruments had symmetrical blades without radial lands or flat sides, similar surface finishing and an almost equiatomic nickel/titanium ratio, whilst the number of spirals, helical angles and the tip geometry were different. PT Ultimate instruments showed 3 distinct heat treatments that matched with the colour of their metal wire. Slider and ProGlider instruments had similar R-phase start (Rs) and R-phase finish (Rf) temperatures. SX, F1, F2, F3 and Shaper instruments showed equivalent heat treatments (Rs ~45.6°C and Rf ~28.3°C) that were similar to their PT Gold counterparts (Rs ~47.9°C and Rf ~28.2°C), but completely distinct to the PT Universal ones (Rs ~16.2°C and Rf ~−18.2°C). Amongst the PT Ultimate instruments, the lowest maximum torques were observed in the SX (0.44 N cm), Slider (0.45 N cm) and Shaper (0.60 N cm) instruments, whilst the highest was noted in the FXL (4.90 N cm). PT Ultimate Slider and ProGlider had similar torsional (~0.40 N cm) and bending loads (~145.0 gf) ($p = 1.000$), whilst the other PT Ultimate instruments showed statistically significantly lower maximum torque, higher angle of rotation and lower bending load (higher flexibility) than their counterparts of the PT Universal and PT Gold systems.

Conclusions: The PT Ultimate system comprises instruments with 3 distinct heat treatments that showed similar phase transformation temperatures to their heat-treated analogues. PT Ultimate instruments presented lower torsional strength and

This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.
© 2022 The Authors. *International Endodontic Journal* published by John Wiley & Sons Ltd on behalf of British Endodontic Society.

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

Tecnologia, Grant/Award Number: UIDB/50025/2020-2023

superior flexibility than their counterparts, whilst maximum torque, angle of rotation and bending loads progressively increased with their sizes.

KEYWORDS

bending load, differential scanning calorimetry, endodontics, nickel-titanium alloy, scanning electron microscope, torsional strength

INTRODUCTION

Nickel-titanium (NiTi) instruments have been widely used to perform the mechanical enlargement of the root canal system. Over several years, successive improvements have been introduced in these instruments including different heat treatments employed during the manufacturing process (Rubio et al., 2022; Zupanc et al., 2018). These changes may lead to distinct crystallographic arrangements of the NiTi alloy at specific temperatures, ultimately influencing the mechanical behaviour of these instruments (Martins et al., 2022).

A few examples of heat-treated alloys are the M-wire (Dentsply Tulsa Dental), which incorporates a heat treatment before the alloy production, and the Gold and Blue heat-treated wires (Dentsply Tulsa Dental) which receive a post-grinding heat treatment (Zupanc et al., 2018). According to Gao et al. (2012), different mechanical behaviours are expected when addressing instruments of similar dimensions manufactured from austenitic NiTi, M-wire or Blue heat-treated alloys. In such cases, M-wire instruments tend to have higher maximum torques, whilst Blue heat-treated wires present lower bending resistance (high flexibility) and higher cyclic fatigue strength and degree of rotation under twist stress (De-Deus et al., 2017; Duke et al., 2015). Likewise, Gold heat-treated instruments usually present superior cyclic fatigue strength and flexibility, but lower torsional strength when compared with conventional (austenitic) NiTi alloy instruments of similar dimensions (Elnaghy & Elsaka, 2016; Plotino et al., 2017). These improvements can be considered relevant in a clinical setup as they may extend the lifespan of instruments, whilst simultaneously preserve the original pathway of the main root canal (Zupanc et al., 2018). Besides, the development of instruments with different features gives to the clinicians the opportunity to choose the most appropriate to a specific root or canal morphology.

Rotary NiTi instruments from the ProTaper (PT) family are probably the most well-known and long-lasting available systems currently on the market. In 2001, when the first generation of this system was launched, instruments were made of conventional NiTi alloy with an innovative design utilizing multiple increasing or decreasing percentage tapers on a single file (Ruddle, 2005). This system originally comprised 3 shaping (SX [19/.04v], S1 [18/.02v] and S2 [20/.04v]) and 3 finishing (F1 [20/.07v], F2 [25/.08v] and F3 [30/.09v]) instruments with sharp cutting edges and no radial lands. Later, 2 larger finishing instruments (F4 [40/.06v] and F5 [50/.05v]) were added to this set and the system changed its name to PT Universal (Dentsply Maillefer). The next generation was launched in 2013, the PT Next (Dentsply Sirona Endodontics), and comprised 5 instruments (sizes 17/.04v, 25/.06v, 30/.07v, 40/.06v and 50/.06v) manufactured in M-wire and designed to have an offset design to improve flexibility and minimize the engagement between the instrument and dentine (Ruddle et al., 2013). Taking advantage of technological advancements in metallurgy, the PT Universal system evolved to PT Gold (Dentsply Sirona Endodontics) in 2014, a system in which instruments have the same geometries, but the alloy is thermomechanically treated (Gold Wire), resulting in an improved flexibility and resistance to cyclic fatigue (Elnaghy & Elsaka, 2016). In this same year, the ProGlider (16/.02v) (Dentsply Sirona Endodontics), an auxiliary rotary instrument that utilizes M-Wire technology, was also introduced for mechanical glidepath preparation (Ruddle et al., 2014).

The novel PT Ultimate rotary system (Dentsply Sirona Endodontics) is the latest generation of the PT family and is one of the first systems to take advantage of distinct crystallographic arrangements induced by specific heat treatment technology to produce a set of instruments with different mechanical behaviours, aiming to ensure a balance between flexibility and strength. According to the manufacturer, the 8 instruments that comprise this system (Slider [16/.02v], SX [20/.03v], Shaper [20/.04v], F1 [20/.07v], F2 [25/.08v], F3 [30/.09v], FX [35/.12v] and FXL [50/.10v]) are manufactured using 3 different heat-treated alloys: M-wire (Slider), Gold-wire (SX, Shaper, F1, F2, F3) and Blue heat-treated wire (FX and FXL) (Dentsply Sirona, 2022). Considering the lack of knowledge regarding this system, a multimethod research approach was conducted to compare the design, metallurgical characteristics and mechanical performance of the PT Ultimate system with instruments of similar sizes from ProGlider, PT Gold and PT Universal systems. The null hypothesis to be tested was that there would be no difference in the mechanical behaviour amongst these different instruments.

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

MATERIAL AND METHODS

Sample selection

A total of 248 new randomly selected NiTi rotary instruments from the novel PT Ultimate (31 instruments of each size – Slider, SX, Shaper, F1, F2, F3, FX, FXL – distributed among design, metallurgical and mechanical assessments) were compared regarding their design, metallurgical characteristics and mechanical behaviour to similar instruments of the ProGlider ($n = 31$), PT Gold ($n = 155$; 31 instruments of each size – SX, S2, F1, F2, F3) and PT Universal ($n = 155$; 31 instruments of each size – SX, S2, F1, F2, F3) systems after being previously checked for major deformations (such as unwinding or major blade discontinuity) that would exclude them from the study. All instruments were 25 mm long, except the SX (19 mm). No major deformation was observed under operative microscope (×13.6) (OPMI Pico; Carl Zeiss Surgical) in any instrument and therefore none of them was excluded.

Design

The microscopic assessment of the design was conducted at ×13.6 magnification (OPMI Pico) in 6 instruments of each size from all tested systems in which the number of blades and the mean helical angles from the 6 most coronal spirals were determined (Image J v1.50e; Laboratory for Optical and Computational Instrumentation). These same instruments were additionally evaluated by scanning electron microscopy (SEM) (Hitachi S-2400; Hitachi) to investigate the symmetry of the blades, the presence of radial lands or flat sides (×20), and the design and type (active or nonactive) of the tips (×40). The surface finishing was also evaluated (×150) regarding the existence of microdefects, such as metal rollovers or spiral discontinuities.

Metallurgy

Energy-dispersive X-ray spectroscopy (EDS) was conducted in 3 instruments of each tested system on a conventional SEM unit (DSM-962 Carl Zeiss Microscopy GmbH) equipped with an Inca X-act EDS detector (Oxford Instruments NanoAnalysis) and set at 20 kV and 3.1 amperes. The initial vacuum was conducted for 10 min, and data acquisition was accomplished in an area of 500 × 400 μm for 1 min at a working distance of 25 mm. Analyses used the ZAF correction and the proportions of metal elements were obtained in a dedicated software (Microanalysis Suite v.4.14 software; Oxford Instruments NanoAnalysis). Differential scanning calorimetry (DSC) tests (DSC 204 F1 Phoenix; NETZSCH-Gerätebau GmbH) were also conducted to determine the phase transformation temperatures (ASTM F2004-17, 2004) using 2 instruments of each size from all tested systems. A fragment of 4–5 mm in length (weighting 5–10 mg) was obtained from the active blade of each instrument and submitted to an etching bath (45% of nitric acid, 25% hydrofluoric acid and 30% of distilled water) for 2 min. After that, the acid solution was neutralized with distilled water and each specimen was mounted on an aluminium pan inside the DSC device, having an empty pan as a control. Each individual thermal cycle had 1 h 40 min duration and ran under gaseous nitrogen (N_2) protection. The cycle temperatures ranged from −150°C to 150°C with a pace of 10°C per minute. The DSC results and charts were obtained using the NETZSCH Proteus Thermal Analysis software (NETZSCH -Gerätebau GmbH). A second test was conducted to confirm the results of the first test.

Mechanical tests

The mechanical behaviour of instruments was evaluated by testing their torsional and bending resistances according to international specifications (ANSI/ADA Specification No. 28, 2002; ISO 3630-3631, 2008). Sample size calculations for the mechanical tests were determined taking into account the highest differences in the results obtained by 2 of the assessed instruments from the PT Ultimate system after 6 initial measurements. Considering an alpha-type error of 0.05 and a power of 80%, the determined sample sizes for maximum torque (effect size: 4.45 ± 2.38; Slider vs. FXL), angle of rotation (effect size: 279.88 ± 162.04; Shaper vs. FXL) and maximum bending load (effect size: 245.42 ± 129.27; Shaper vs. FX) were 6, 7 and 6 instruments, respectively. The final sample size for each test was set as 10 instruments for all groups.

In the torsional test, instruments were mounted in a straight position on a torsiometer (TT100; Odeme Dental Research) and clamped at their apical 3 mm. Then, they were rotated on a constant pace of 2 rpm in a clockwise direction until fracture. The maximum torque sustained prior to rupture (in N cm) and the angle of rotation (in degrees) were assessed with a dedicated software (Odeme Analysis TT100, Odeme Dental Research). In the bending test, instruments were mounted in the file holder and positioned at 45° in relation to the floor, whilst their apical 3 mm were attached to a wire connected to a universal testing machine (DL-200 MF; EMIC). The test was conducted using a 20 N load applied at a 15 mm/min constant pace until the instrument

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

accomplished a 45° displacement. The maximum load required to induce this displacement was recorded in gram/force (gf) using the Tesc v3.04 software (Mattest Automação e Informática).

Statistical analysis and reporting

Data normality was assessed using the Shapiro-Wilk test and presented as mean (standard deviation) or median (interquartile range) depending on their distribution. One-way ANOVA *post hoc* Tukey tests were used to assess differences in the mean helical angles, whilst the nonparametric Mood's median test was employed to compare maximum torque, angle of rotation and maximum bending load amongst instruments (SPSS v22.0 for Windows; SPSS Inc.). The level of significance was set at 5%. The present manuscript was written according to Preferred Reporting Items for Laboratory studies in Endodontology (PRILE) 2021 guidelines (Figure 1) (Nagendrababu et al., 2021).

RESULTS

Design

Table 1 summarizes the analyses of design, whilst Figure 2 shows the SEM images of the assessed instruments. All tested files had symmetrical blades without radial lands or flat sides.

PT Ultimate Slider was similar to ProGlider in terms of tip size, surface finishing and helical angle, but had a shorter active area with a smaller number of blades and a parallelogram cross-section, whilst the ProGlider had a square horizontal cross-section. The number of blades of the PT Ultimate Shaper and Finishers (F1, F2 and F3) decreased (from 18 to 12), as the diameter increased, and was higher than their counterparts, whose spirals also decreased from 11 (S2) to 9 (F3). Overall, helical angles were similar amongst instruments, however PT Ultimate F1 and F2 showed significant lower angles than their equivalent PT Universal and PT Gold instruments (Table 1). PT Ultimate Shaper and Finishers had an off-centred parallelogram cross-section, whilst all analogue instruments had a convex cross-sectional triangular shape, except for the F3 instruments that had a concave triangular cross-section. PT Ultimate FX and FXL had the smallest number of blades and helical angles amongst tested systems, but similar cross-sections to the other PT Ultimate instruments. The tips of the PT Ultimate Shaper and Finishers were similar, but different from the Slider, FX and FXL, whilst in the other systems, the geometry of the tips was distinct from each other. None of the tips could be clearly identified as active.

Visual and microscopic analyses of all instruments revealed no major deformations or defects. In general, surface finishing was similar with manufacturing parallel marks in all instruments and only very few micro defects.

Metallurgy

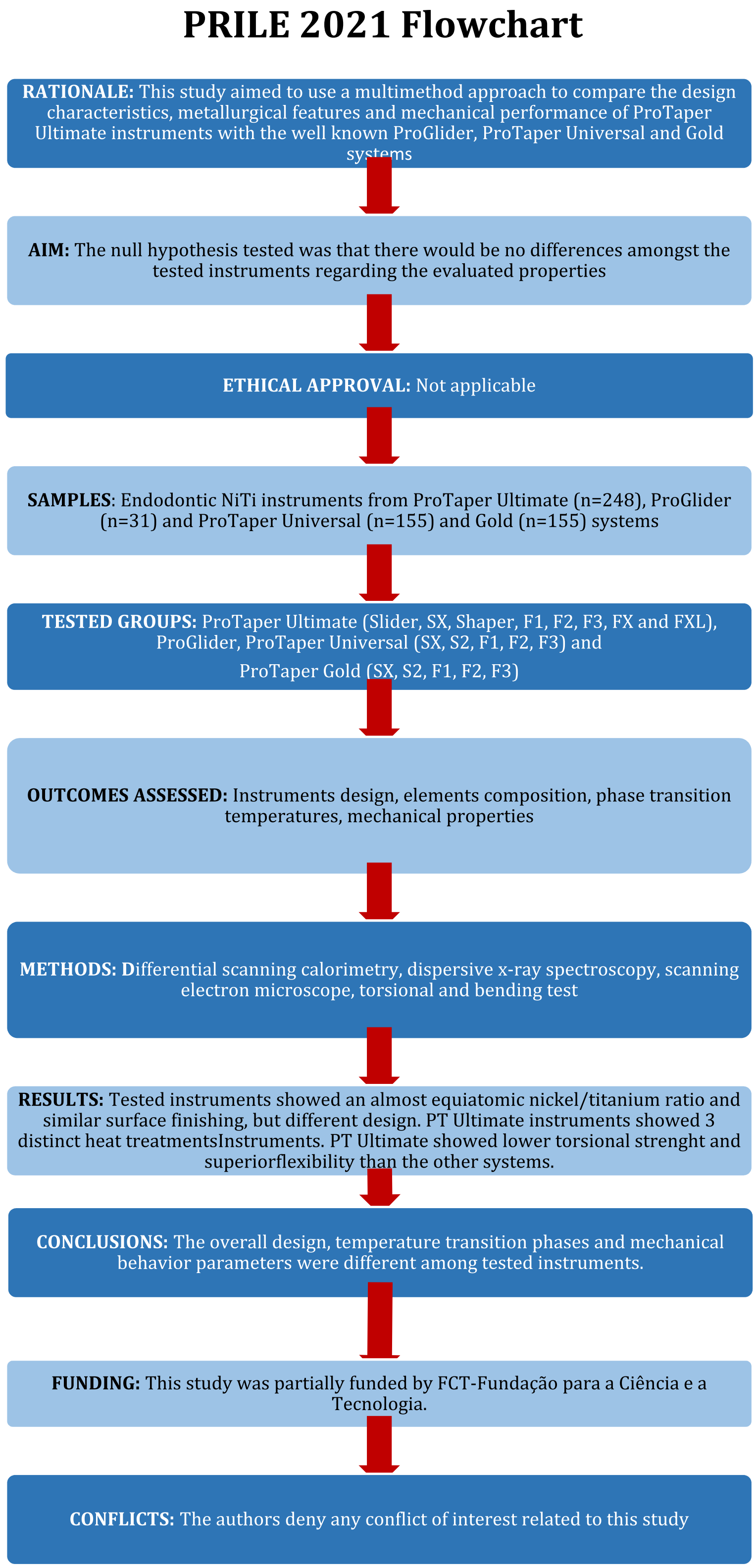
Energy-dispersive X-ray spectroscopy tests showed an almost equiatomic nickel/titanium elements ratio in all instruments with no other metal element. DSC analyses of the 8 instruments of the PT Ultimate system revealed 3 distinct heat treatments that matched with the colour of their metal alloy (Figure 3). The Slider and the ProGlider instruments had similar R-phase start (Rs) and R-phase finish (Rf) temperatures. SX, F1, F2, F3 and Shaper instruments showed equivalent heat treatments (Rs ~45.6°C and Rf ~28.3°C) that were similar to their PT Gold counterparts (Rs ~47.9°C and Rf ~28.2°C), but completely distinct to the PT Universal ones (Rs ~16.2°C and Rf ~−18.2°C). PT Ultimate FX and FXL instruments showed similar DSC curves with phase transformation temperatures ranging from 29.4°C (Rs) and 19.8°C (Rf) at cooling, and 7.7°C (austenitic start [As]) and 36.4°C (austenitic finish [Af]) at heating (Table 2, Figure 3).

Mechanical tests

Amongst the PT Ultimate instruments, the lowest maximum torques were observed in the SX (0.44 N cm), Slider (0.45 N cm) and Shaper (0.60 N cm) instruments, whilst the highest was noted in the FXL (4.90 N cm) (Table 1). The lowest and highest angles of rotation were observed in the Shaper (418°) and FXL (712°) instruments, respectively. Although the bending test revealed an overall tendency of instruments to become less flexible as they increased in size, the largest instrument of this system (FXL) showed a maximum load significantly lower (294.4 gf) than the FX instrument (410.9 gf), which was the least flexible amongst the 25-mm instruments (Table 1, Figure 4). PT Ultimate Slider and ProGlider had similar torsional ($p = 1.000$) and bending load ($p = 1.000$) results, whilst, in general, the other PT Ultimate instruments showed statistically significantly lower maximum torque, higher angle of rotation and lower bending load (higher flexibility) than their counterparts of the PT Universal and PT Gold systems (Table 1, Figure 4).

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

FIGURE 1 PRILE (2021) flowchart (Nagendrababu et al., 2021).



DISCUSSION

This study presents original data regarding the recently launched PT Ultimate file-specific heat-treated rotary system using the multimethod research concept, an approach that provides more information, better understanding and superior internal and external validation than a single or double method assessment (Martins et al., 2021c). Overall, the concept of the PT Ultimate system seems to combine several features from previous instruments developed by the same company including the variable taper (ProTaper), the so-called 'Deep Shape' concept or

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

TABLE 1 Design characteristics and mechanical test results of ProTaper (PT) Ultimate, Gold, Universal and ProGlider instruments expressed as mean (standard deviation) or median [interquartile range]

	Design		Torsional test		Bending test
Instruments (tip size/taper, wire)	Number of blades	Helical angle (°)	Maximum torque (N cm)	Angle of rotation (°)	Maximum load (gf)
PT Ultimate Slider (16/.02v, M-Wire)	19	21.4 (±1.9)	0.45 [0.38–0.50]	435.4 [404.9–478.7]	149.7 [139.9–176.9]
ProGlider (16/.02v, M-Wire)	21	21.2 (±0.8)	0.40 [0.30–0.50]	429.0 [419.3–463.0]	144.8 [136.9–149.6]
PT Ultimate SX (20/.03v, Gold)	12	16.9 (±1.7)	0.44 [0.38–0.50][a]	422.1 [343.3–507.0]	538.3 [511.9–570.4][a]
PT Gold SX (19/.04v, Gold)	9	19.9 (±3.3)	0.55 [0.48–0.63][b]	387.6 [330.0–455.5]	490.1 [471.5–504.9][b]
PT Universal SX (19/.04v, Austenitic)	9	20.6 (±3.0)	0.31 [0.20–0.40][a]	405.3 [384.8–435.8]	876.0 [833.3–926.9][c]
PT Ultimate Shaper (20/.04v, Gold)	18	21.9 (±2.9)	0.60 [0.48–0.73][a]	408.9 [364.2–485.3][a]	229.1 [208.9–236.4][a]
PT Gold S2 (20/.04v, Gold)	11	22.1 (±1.2)	1.00 [0.88–1.20][b]	506.5 [471.0–542.8][b]	255.1 [241.6–260.7][b]
PT Universal S2 (20/.04v, Austenitic)	11	22.4 (±2.5)	0.70 [0.58–0.80][a]	408.0 [362.8–455.3][a]	546.9 [524.5–574.6][c]
PT Ultimate F1 (20/.07v, Gold)	16	18.4 (±2.9)[a]	1.05 [1.00–1.20][a]	478.9 [417.2–584.9][a]	200.9 [188.7–210.7][a]
PT Gold F1 (20/.07v, Gold)	12	25.3 (±1.1)[b]	1.30 [1.20–1.40][b]	486.0 [447.5–534.8][a]	261.0 [237.7–274.5][b]
PT Universal F1 (20/.07v, Austenitic)	12	25.6 (±0.8)[b]	1.30 [1.08–1.40][a,b]	359.5 [304.3–390.8][b]	393.3 [389.7–406.8][c]
PT Ultimate F2 (25/.08v, Gold)	16	19.1 (±1.5)[a]	1.40 [1.30–1.40][a]	489.1 [449.9–618.2][a]	204.1 [192.8–219.5][a]
PT Gold F2 (25/.08v, Gold)	10	21.9 (±1.3)[b]	1.50 [1.40–1.53][b]	414.0 [390.0–452.3][b]	249.4 [241.2–251.1][b]
PT Universal F2 (25/.08v, Austenitic)	10	22.4 (±1.4)[b]	1.80 [1.58–1.83][c]	310.0 [289.0–329.0][c]	494.1 [485.6–509.3][c]
PT Ultimate F3 (30/.09v, Gold)	12	18.7 (±2.2)	1.45 [1.28–2.13][a]	632.1 [457.6–791.6][a]	254.9 [240.2–272.3][a]
PT Gold F3 (30/.09v, Gold)	9	20.7 (±3.8)	1.70 [1.60–1.85][a,b]	639.0 [618.0–711.8][a]	279.7 [269.5–304.4][a]
PT Universal F3 (30/.09v, Austenitic)	9	20.9 (±4.6)	2.10 [1.80–2.50][b]	469.5 [438.0–481.0][b]	681.7 [661.5–698.1][b]
PT Ultimate FX (35/.12v, Blue)	8	18.1 (±3.4)	3.35 [1.38–3.83]	659.9 [486.3–746.1]	416.1 [397.4–428.8]
PT Ultimate FXL (50/.10v, Blue)	5*	19.0 (±2.0)	4.90 [4.53–5.23]	712.3 [645.1–807.4]	294.4 [290.8–301.4]

Note: Different letters in the same column related to the same group of analogue instruments mean statistically significant differences ($p < .05$).

*Only 5 blades were measured instead of 6.

increased apical taper (ProTaper), the off-centred parallelogram cross-section (PT Next, TruNatomy), the large-tapered FXL auxiliary instrument (ProFile GT) and the use of M-Wire (ProGlider, PT Next), Gold wire (PT Gold, WaveOne Gold) and Blue wire (Vortex Blue, Reciproc Blue) heat-treated metal alloys. Amongst the PT Ultimate instruments, it was observed that the maximum torque sustainable prior to fracture and maximum bending loads increased with instruments' size (Table 1, Figure 4), an expected result considering previous studies on multi-file systems reporting higher torques and less flexibility in larger instruments (Kramkowski & Bahcall, 2009; Ninan & Berzins, 2013; Pedulla et al., 2018; Viana et al., 2010; Wycoff & Berzins, 2012). In contrast, no pattern could be demonstrated in the angle of rotation according to instruments' size, but mixed results in this mechanical parameter have been also reported by several authors (Kramkowski & Bahcall, 2009; Ninan & Berzins, 2013; Pedulla et al., 2018; Wycoff & Berzins, 2012). The different crystallographic arrangements of PT Ultimate instruments, however, did not seem to influence their mechanical behaviour since these results could be mostly explained by differences in the dimensions of instruments. An exception was observed in the largest instrument of the PT Ultimate system, the FXL (50/.10v), which was more flexible than the FX (35/.12v), an instrument made with the same heat treatment alloy, but with small dimensions (Table 1, Figure 3). This apparent contradictory result may be explained considering that the active part of the FXL has only 7 mm in length, and, therefore, the result of the bending test reflected the cross-sectional diameter of its nonactive portion, which is smaller (1 mm) than the FX instrument (1.2 mm at D16).

The mechanical performance of the tested instruments can be partially explained by the dissimilarities observed

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

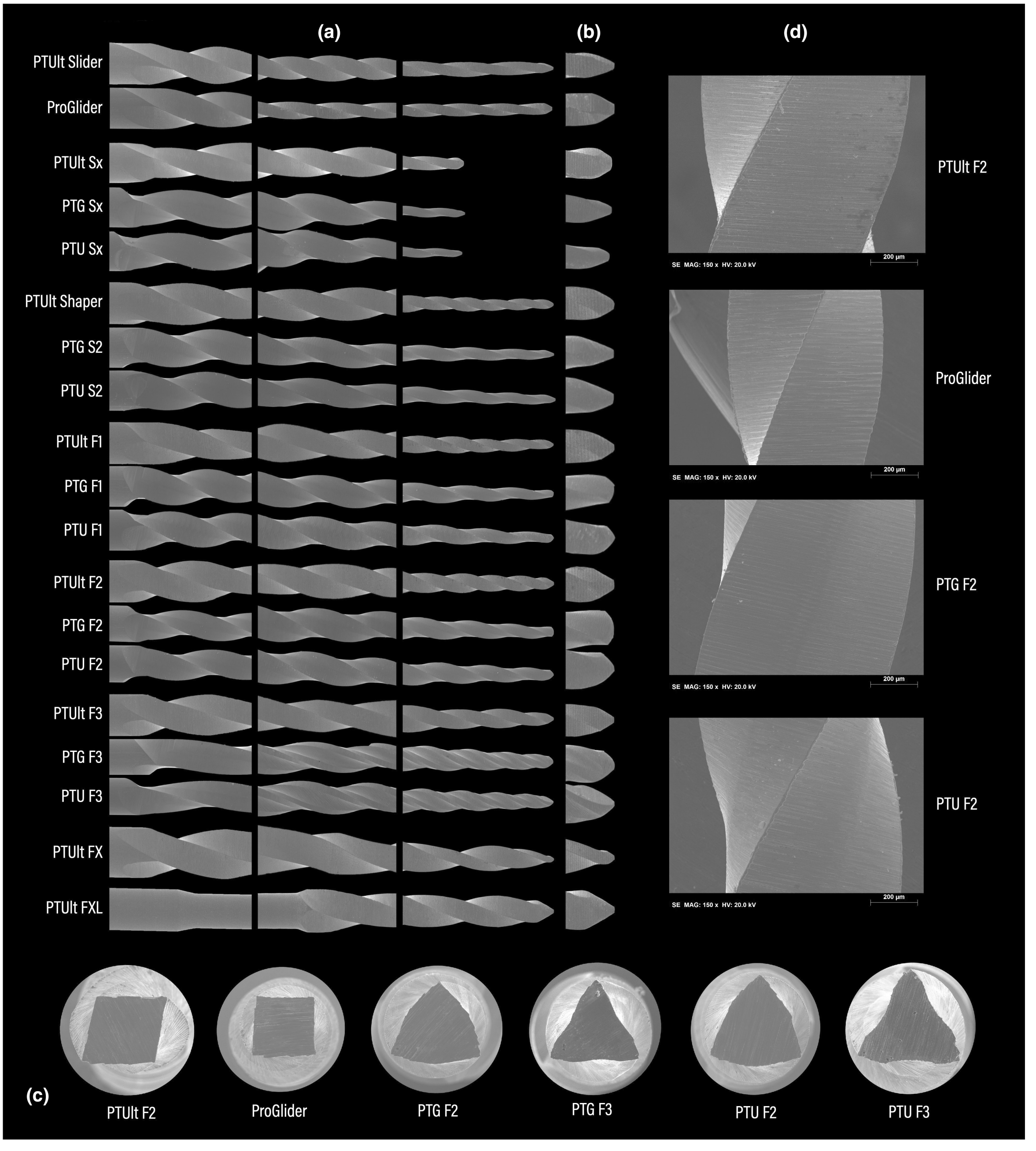


FIGURE 2 Scanning electron microscopic analyses of ProGlider, ProTaper Ultimate (PTUlt), ProTaper Gold (PTG) and ProTaper Universal (PTU) instruments. Representative images of the (a) active blades (×20), (b) tips (×40), (c) cross-sections (at D10) (×80) and (d) surface finishing (×150). Except for the FXL, the design of the other PTUlt instruments was similar, but different from their equivalent counterparts. Surface finishing (d) was similar amongst instruments with the presence of parallel manufacturing mark and few micro defects.

in their geometry, mostly because changes in the design of the novel PT Ultimate system do not allow to compare one-to-one with the old versions of ProTaper instruments, highlighting the importance of a multimethod analysis to properly understand their mechanical behaviour. The present results demonstrated that PT Ultimate Shaper and Finishers (F1, F2 and F3) had lower torsional strength and superior flexibility (higher angle of rotation and lower bending load) compared with their counterparts (Table 1, Figure 4) and the null hypothesis was rejected. Considering the similarities of tested instruments in terms of nickel/titanium ratio and surface finishing, results of these PT Ultimate instruments may be mostly explained not only by their different designs, such as the high number of spirals (McSpadden, 2007) (Table 1) and the off-centred parallelogram cross-section (Martins et al., 2020) (Figure 2), but also by their crystallographic arrangement compared to the full austenitic PT Universal, once the alloy of the PT

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

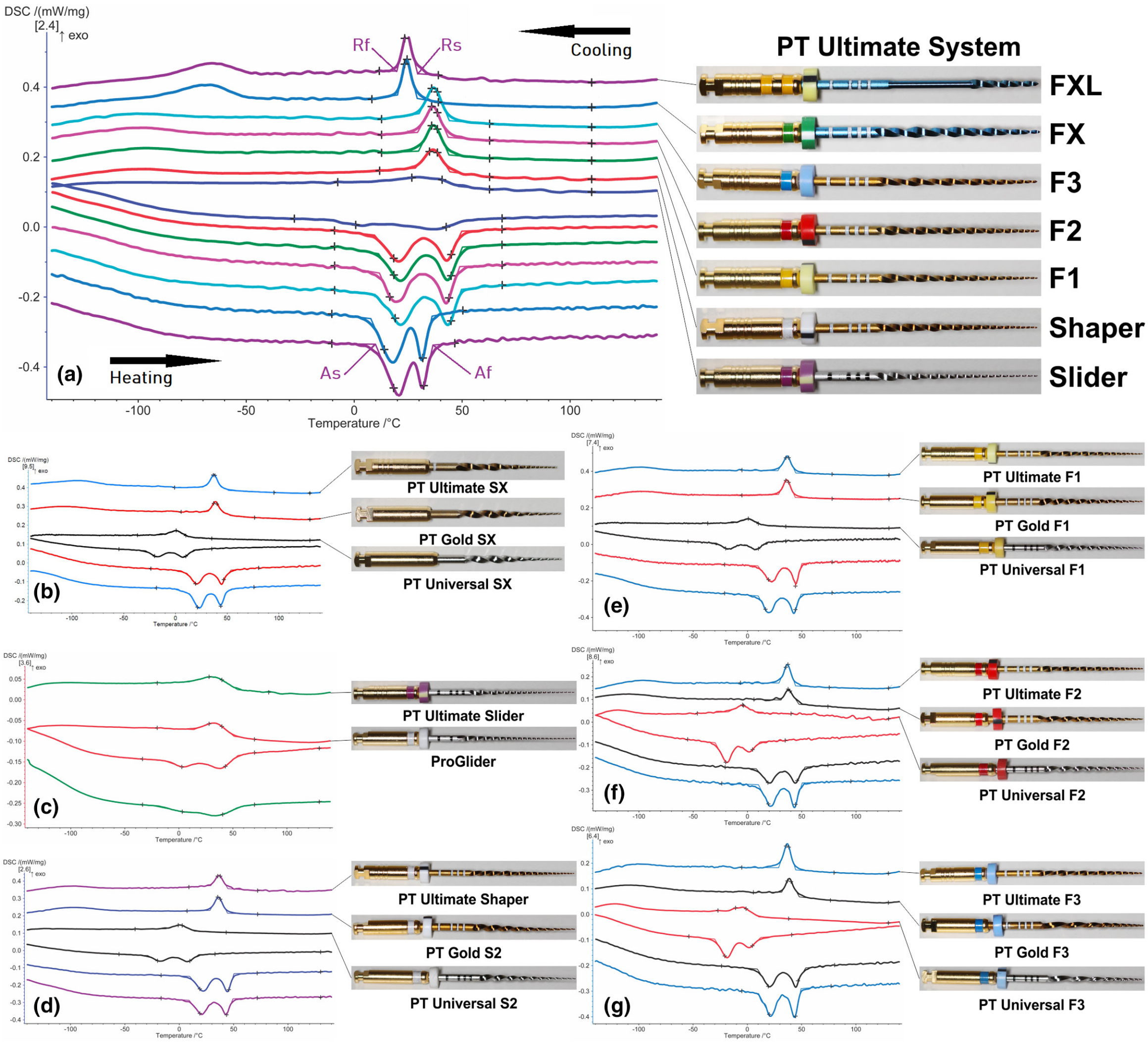


FIGURE 3 Comparison of DSC curves and phase transformation temperatures amongst the ProTaper (PT) Ultimate (a) instruments and their equivalent counterparts (b–g), depicting their macroscopic views in which differences in their alloy colours suggest distinct heat treatments. (a) The analyses of the PT Ultimate system revealed 3 different curve patterns: (1) Slider had a unique curve with the highest and lowest phase transformation temperatures at both cooling (top curve, reading from right to left) and heating (bottom curve, reading from left to right); (2) SX, Shaper, F1, F2 and F3 instruments share similar DSC curves (Rs ~44.0°C; Rf ~29.0°C), as well as, (3) FX and FXL instruments on cooling (Rs ~29.0°C; Rf ~20°C) and heating (As 7.7–9.8°C; Af ~36.0°C). (b–g) PT Ultimate instruments showed equivalent DSC curves and phase transformation temperatures to their counterparts, except for the ProTaper Universal instruments. (As Austenitic start; Af Austenitic finish; Rs R-phase start; Rf R-phase finish).

Gold system has similar heat treatment (Figure 3, Table 2). Compared with the other tested instruments, the reduced flexibility of the SX instruments (Table 1, Figure 4) may be related to their shorter lengths (19 mm) which led to an exponential increase in the stress necessary to apply the force during the standardized bending test.

NiTi alloys may have three distinct microstructural phases named austenite, R-phase and martensite, which can directly influence the mechanical behaviour of endodontic instruments (Elnaghy & Elsaka, 2016; Plotino et al., 2017; Zupanc et al., 2018). The austenitic phase of the NiTi alloy is relatively stiff, hard and has limited flexibility. When stress is applied to this type of instrument, a transformation from the austenitic to the martensitic crystallographic arrangement may occur in a process named stress-induced martensitic transformation This atomic reorganization leads to a feature known as superelasticity, characterized by a form rearrangement that may spring back the instrument to its original form without any definitive deformation when the induced stress is stopped or reduced (Shen et al., 2011), meaning that its lower elastic modulus, compared with stainless-steel instruments, provides superior flexibility (Zupanc et al., 2018). The austenitic form, and its superelasticity features, characterizes the NiTi conventional alloy that has been used in systems such as the ProTaper Universal tested in this study.

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

TABLE 2 Phase transformation temperatures (in °C) of ProTaper (PT) Ultimate, Gold, Universal and ProGlider instruments

Instrument (tip size/taper, wire)	R-Phase start (Rs)	R-Phase finish (Rf)	Austenitic start (As)	Austenitic finish (Af)
PT Ultimate Slider (16/.02v, M-Wire)	48.4	11.5	−13.3	52.1
ProGlider (16/.02v, M-Wire)	51.2	13.9	−14.4	54.6
PT Ultimate SX (20/.03v, Gold)	45.6	29.1	11.5	50.6
PT Gold SX (19/.04v, Gold)	47.7	31.6	9.6	54.1
PT Universal SX (19/.04v, Austenitic)	16.2	−13.5	−28.9	17.9
PT Ultimate Shaper (20/.04v, Gold)	44.3	28.3	9.4	49.7
PT Gold S2 (20/.04v, Gold)	44.4	28.2	9.6	50.4
PT Universal S2 (20/.04v, Austenitic)	14.1	−12.5	−28.9	18.0
PT Ultimate F1 (20/.07v, Gold)	43.6	29.3	11.7	48.9
PT Gold F1 (20/.07v, Gold)	43.9	28.4	9.4	50.0
PT Universal F1 (20/.07v, Austenitic)	16.2	−13.3	−28.7	17.6
PT Ultimate F2 (25/.08v, Gold)	43.9	29.8	11.2	50.1
PT Gold F2 (25/.08v, Gold)	47.5	31.0	8.1	53.1
PT Universal F2 (25/.08v, Austenitic)	9.8	−17.7	−29.8	11.7
PT Ultimate F3 (30/.09v, Gold)	44.0	30.1	9.6	49.7
PT Gold F3 (30/.09v, Gold)	47.9	31.7	9.3	53.8
PT Universal F3 (30/.09v, Austenitic)	10.7	−18.2	−30.1	12.4
PT Ultimate FX (35/.12v, Blue)	29.2	20.0	7.7	36.1
PT Ultimate FXL (50/.10v, Blue)	29.4	19.8	9.8	36.4

The crystallographic arrangement of the NiTi alloy observed in a higher temperature range is defined as the austenitic phase and is characterized by a B2 type lattice (cubic symmetry). When the alloy temperature decreases below the transformation temperature range, the martensitic transformation occurs from the austenitic phase to the martensitic one. This martensitic phase displays a monoclinic lattice (B19′ type) that can be reverted to the B2 type lattice by heating the alloy above the transformation temperature range (Thompson, 2000). This phenomenon of changing the physical properties to allow a deformed NiTi alloy recovers its original shape when heated is known as shape memory (Zupanc et al., 2018). Companies take advantage of this property to produce martensitic instruments that are heat treated during their manufacture to raise their phase transformation temperatures. As a result, these instruments are softer, more ductile and have superior flexibility, cyclic fatigue resistance and lower strength to torsional stress than instruments with austenitic crystallographic arrangements. Several designations have been given to these heat-treated NiTi alloys, such as M-wire, CM wire, Gold wire, Blue wire, or MaxWire (Zupanc et al., 2018). Notwithstanding

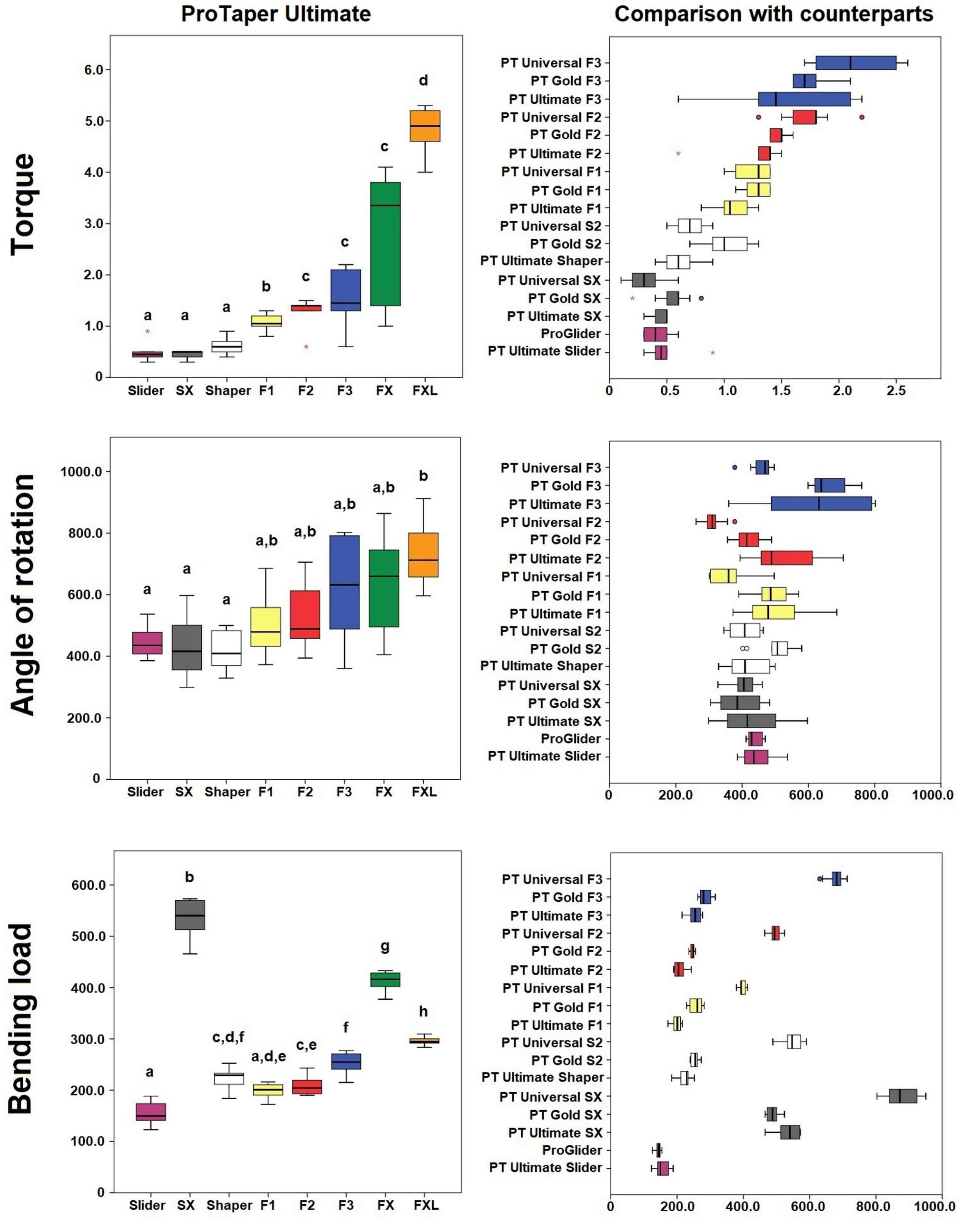


FIGURE 4 Comparison of the mechanical behaviour amongst the ProTaper (PT) Ultimate instruments and their equivalent counterparts. (On the left column) In the PT Ultimate system, the maximum torque, angle of rotation and bending load progressively increased with instruments' sizes, except for the FXL that was more flexible than the FX instrument. (On the right column) PT Ultimate showed lower torsional strength, superior flexibility and higher angle of rotation than PT Gold and PT Universal counterparts. Slider and ProGlider had similar mechanical behaviour. Different letters in the charts represent statistically significant differences ($p < 0.05$).

the fact that all of them share similar martensitic characteristics, they have distinct crystallographic arrangements at service temperature and, consequently, different mechanical behaviours (Zupanc et al., 2018), as depicted by the present results (Table 1, Figures 3 and 4). Another type of martensitic transformation, which occurs between full austenitic and full martensitic forms, is the R-phase transformation, which may also be considered a martensitic

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

form (Kuhn & Jordan, 2002). It consists of a rhombohedral atomic disposition with thermoelastic martensitic characteristics and, similarly to the martensitic phase, can be stress- or temperature-induced. Many manufacturers have used this R-phase transformation to produce instruments with some ductility, but enhanced flexibility and cyclic fatigue strength, compared to conventional NiTi instruments (Zhou et al., 2013; Zupanc et al., 2018).

One of the innovations of the PT Ultimate system was the file-specific heat treatment based on instruments' dimensions featuring M-wire (Slider), Gold (SX, Shaper and Finishers F1, F2 and F3) and Blue (Auxiliary Finishers FX and FXL) heat-treated wires, that is, instruments presenting 3 distinct crystallographic arrangements of their metal alloys (mixed austenitic, R-phase and martensitic forms depending on the temperature of the instrument) in the same system, a feature confirmed in this study (Figure 3). The idea behind this approach is to take advantage of different crystallographic phases of the NiTi alloy to create instruments with enhanced properties according to their use requirements. The Slider and ProGlider instruments showed equivalent DSC curves that were consistent to M-wire instruments (Martins et al., 2021a; Martins et al., 2021b), but distinct to the other instruments of the PT Ultimate system (Table 2, Figure 3). The Slider has an austenitic plus R-phase crystallographic arrangement at both room and body temperatures and, therefore, minor changes in its mechanical behaviour may be expected in that service temperature range. The Shaper and Finishers (F1, F2 and F3) of the PT Ultimate system appear to present a martensitic crystallographic arrangement at room temperature after manufacturing and tend to acquire a mixed austenitic plus R-phase characteristics when enclosing the body temperature meaning that, at higher temperatures, instruments may develop some characteristics of the austenitic alloy. These instruments present a R-phase transformation at cooling (between 44.3°C [Rs] and 28.3°C [Rf]) with a transition to B19' at a very low temperature (under −50°C) but with a DSC double curve from B19' to R-phase to B2 on heating in a more proximal temperature range (between 9.4°C and 50.1°C) (Figure 3). These transformation temperatures were similar to their analogues PT Gold instruments, but distinct from the PT Universal ones (Table 2, Figure 3), and followed previous reports testing gold wire instruments (Martins et al., 2021b).

The auxiliary FX and FXL instruments of the PT Ultimate system showed DSC curves and phase transformation temperatures between 29.4°C (Rs) and 19.8°C (Rf) on cooling and 7.7°C (As) and 36.4°C (Af) on heating (Table 2), corroborating with previous studies testing Blue heat-treated wire instruments (Martins et al., 2021b). These 2 instruments present a martensitic crystallographic arrangement at room temperature, which tends to change to an austenitic form at body temperature. Therefore, the incorporation of more austenitic characteristics in these instruments is expected if their temperature rises during root canal preparation procedures, decreasing their flexibility (Oh et al., 2020) and their ability to sustain high maximum torque (Silva et al., 2018). These results, however, raise doubts regarding the decision of the manufacturer to use the Blue heat-treated wire in the FX and FXL auxiliary instruments. One possible argument would be the intention of increasing their austenite phase, consequently improving their torsional strength resistance. But this makes no sense since both instruments are recommended to be used only in anatomically straight and large canals that were previously enlarged by the other instruments (Ruddle, 2022), a condition in which they are submitted only to a low torsional stress. Therefore, a proper explanation from the manufacturer regarding the advantage of using the Blue heat-treated wire in these auxiliary instruments is still missing. Considering that PT Gold and PT Universal systems do not have instruments with similar dimensions of FX and FXL, no comparisons with other instruments could be made.

The main limitations of this study include not evaluating parameters such as cyclic fatigue strength, cutting efficiency and shaping ability, which should be included in future studies. Besides, it was also not possible to determine the real influence of the different cross-sections in the mechanical properties of tested instruments. On the other hand, the major strengths were to provide essential information about the design, metallurgy and mechanical behaviour of the recently launched PT Ultimate, a system that comprises instruments with specific heat treatments and distinct crystallographic arrangements of their metal alloys, through a multimethod research using well-established international guidelines (ANSI/ADA Specification No. 28, 2002; ASTM F2004-17, 2004; ISO 3630-3631, 2008). This methodological approach allows for a more comprehensive understanding of the results since it overcomes the inherent limitations of each test. Considering that the novel PT Ultimate system showed lower torsional strength and higher flexibility than their counterparts, clinicians may benefit from this system in clinical cases that requires these characteristics, such as curved and nonconstricted root canals, instead of PT Universal or PT Gold; however, considering the lack of information about this recently launched system, further studies are still needed to guide clinical recommendations.

CONCLUSIONS

The novel PT Ultimate system comprises instruments with three distinct heat treatments that showed different

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

design, but similar surface finishing, nickel/titanium ratios and phase transformation temperatures to their heat-treated analogues. Whilst Slider and ProGlider had similar mechanical behaviour, the other PT Ultimate instruments showed lower torsional strength and superior flexibility than their counterparts, whilst maximum torque, angle of rotation and bending loads progressively increased with their sizes.

AUTHOR CONTRIBUTIONS

J.N.R. Martins: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Writing – original draft presentation. E.J.N.L. Silva: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Writing – original draft presentation. D. Marques: Data curation; Methodology; Supervision; Writing – review & edit. N. Ajuz: Investigation; Methodology; Validation. M. Rito Pereira: Investigation, Resources; Writing – review & edit. R. Pereira da Costa: Data curation, Resources; Writing – review & edit. F.M. Braz Fernandes: Data curation; Formal analysis; Funding acquisition; Writing – review & edit. M.A. Versiani: Conceptualization; Methodology; Supervision; Writing – review & edit.

ACKNOWLEDGEMENTS

FMBF acknowledges the funding of CENIMAT/i3N by national funds through the FCT-Fundação para a Ciência e a Tecnologia, I.P., within the scope of Multiannual Financing of R&D Units, reference UIDB/50025/2020-2023.

CONFLICT OF INTEREST

The authors deny any conflicts of interest.

DATA AVAILABILITY STATEMENT

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

ORCID

Jorge N. R. Martins https://orcid.org/0000-0002-6932-2038
Emmanuel João Nogueira Leal Silva https://orcid.org/0000-0002-6445-8243
Marco Aurélio Versiani https://orcid.org/0000-0001-5277-9827

REFERENCES

ANSI/ADA Specification No. 28-2002. (2002) *Root canal files and reamers, type K for hand use.*

ASTM F2004-17. (2004) *ASTM International. Standard test method for transformation temperature of nickel-titanium alloys by thermal analysis*:1–5.

De-Deus, G., Silva, E.J., Vieira, V.T., Belladonna, F.G., Elias, C.N., Plotino, G. et al. (2017) Blue thermomechanical treatment optimizes fatigue resistance and flexibility of the Reciproc files. *Journal of Endodontics*, 43, 462–466.

Dentsply Sirona. *ProTaper Ultimate Endodontic Files [WWW document]*. Available from: https://www.dentsplysirona.com/en-gb/categories/endodontics/protaper-ultimate-files.html [Accessed 13th July 2022].

Duke, F., Shen, Y., Zhou, H., Ruse, N.D., Wang, Z.J., Hieawy, A. et al. (2015) Cyclic fatigue of ProFile Vortex and Vortex Blue nickel-titanium files in single and double curvatures. *Journal of Endodontics*, 41, 1686–1690.

Elnaghy, A.M. & Elsaka, S.E. (2016) Mechanical properties of ProTaper Gold nickel-titanium rotary instruments. *International Endodontic Journal*, 49, 1073–1078.

Gao, Y., Gutmann, J.L., Wilkinson, K., Maxwell, R. & Ammon, D. (2012) Evaluation of the impact of raw materials on the fatigue and mechanical properties of ProFile Vortex rotary instruments. *Journal of Endodontics*, 38, 398–401.

ISO 3630-3631. (2008) Dentistry – Root canal instruments – Part 1: General requirements and test methods.

Kramkowski, T.R. & Bahcall, J. (2009) An in vitro comparison of torsional stress and cyclic fatigue resistance of ProFile GT and ProFile GT Series X rotary nickel-titanium files. *Journal of Endodontics*, 35, 404–407.

Kuhn, G. & Jordan, L. (2002) Fatigue and mechanical properties of nickel-titanium endodontic instruments. *Journal of Endodontics*, 28, 716–720.

Martins, J.N.R., Silva, E., Marques, D., Belladonna, F., Simoes-Carvalho, M., Camacho, E. et al. (2021a) Comparison of design, metallurgy, mechanical performance and shaping ability of replica-like and counterfeit instruments of the ProTaper Next system. *International Endodontic Journal*, 54, 780–792.

Martins, J.N.R., Silva, E., Marques, D., Belladonna, F., Simoes-Carvalho, M., Vieira, V.T.L. et al. (2021b) Design, metallurgical features, mechanical performance and canal preparation of six reciprocating instruments. *Interational Endodontic Journal*, 54, 1623–1637.

Martins, J.N.R., Silva, E., Marques, D., Pereira, M.R., Arantes-Oliveira, S., Martins, R.F. et al. (2021c) Evaluation of design, metallurgy, microhardness, and mechanical properties of glide path instruments: a multimethod approach. *Journal of Endodontics*, 47, 1917–1923.

Martins, J.N.R., Silva, E., Marques, D., Pereira, M.R., Vieira, V.T.L., Arantes-Oliveira, S. et al. (2022) Design, metallurgical features, and mechanical behaviour of NiTi endodontic instruments from five different heat-treated rotary systems. *Materials*, 15, 1009.

Martins, S., Garcia, P., Viana, A., Buone, V. & Santos, L. (2020) Off-centered geometry and influence on NiTi endodontic file performance evaluated by finite element analysis. *Journal of Materials Engineering and Performance*, 29, 2095–2102.

McSpadden, J.T. (2007) Mastering instrument designs. In: McSpadden, J.T. (Ed.) *Mastering endodontics instrumentation*. Chattanooga, USA: Cloudland Institute, pp. 37–97.

Nagendrababu, V., Murray, P.E., Ordinola-Zapata, R., Peters, O.A., Rôças, I.N., Siqueira, J.F., Jr. et al. (2021) PRILE 2021 guidelines for reporting laboratory studies in Endodontology: explanation and elaboration. *International Endodontic Journal*, 54, 1491–1515.

Ninan, E. & Berzins, D.W. (2013) Torsion and bending properties of shape memory and superelastic nickel-titanium rotary instruments. *Journal of Endodontics*, 39, 101–104.

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

Oh, S., Kum, K.Y., Kim, H.J., Moon, S.Y., Kim, H.C., Chaniotis, A. et al. (2020) Bending resistance and cyclic fatigue resistance of WaveOne Gold, Reciproc Blue, and HyFlex EDM instruments. *Journal of Dental Sciences*, 15, 472–478.

Pedulla, E., Lo Savio, F., La Rosa, G.R.M., Miccoli, G., Bruno, E., Rapisarda, S. et al. (2018) Cyclic fatigue resistance, torsional resistance, and metallurgical characteristics of M3 Rotary and M3 Pro Gold NiTi files. *Restorative Dentistry & Endodontics*, 43, e25.

Plotino, G., Grande, N.M., Mercade Bellido, M., Testarelli, L. & Gambarini, G. (2017) Influence of temperature on cyclic fatigue resistance of ProTaper Gold and ProTaper Universal rotary files. *Journal of Endodontics*, 43, 200–202.

Rubio, J., Zarzosa, J., Aranda, S., Casino, A. & Pallarés, A. (2022) A comparative study of cyclic fatigue of 6 endodontic systems. An in vitro study. *Journal of Clinical and Experimental Dentistry*, 14, e560–e565.

Ruddle, C.J. (2005) The ProTaper technique. *Endodontic Topics*, 10, 187–190.

Ruddle, C.J. (2022) *The Ultimate shaping system: an opening for 3D cleaning and filling root canals [WWW document]*. Available from: https://www.oralhealthgroup.com/features/the-ultimate-shaping-system-an-opening-for-3d-cleaning-and-filling-root-canals/ [Accessed 7th September 2022]

Ruddle, C.J., Machtou, P. & West, J.D. (2013) The shaping movement: fifth-generation technology. *Dentistry Today*, 32, 94–99.

Ruddle, C.J., Machtou, P. & West, J.D. (2014) Endodontic canal preparation: innovations in glide path management and shaping canals. *Dentistry Today*, 33, 118–123.

Shen, Y., Zhou, H.M., Zheng, Y.F., Campbell, L., Peng, B. & Haapasalo, M. (2011) Metallurgical characterization of controlled memory wire nickel-titanium rotary instruments. *Journal of Endodontics*, 37, 1566–1571.

Silva, E., Hecksher, F., Antunes, H.D.S., De-Deus, G., Elias, C.N. & Vieira, V.T.L. (2018) Torsional fatigue resistance of Blue-treated reciprocating instruments. *Journal of Endodontics*, 44, 1038–1041.

Thompson, S.A. (2000) An overview of nickel-titanium alloys used in dentistry. *International Endodontic Journal*, 33, 297–310.

Viana, A.C., Craveiro, C., de Melo, M., de Azevedo, G., Bahia, M. & Lopes Buono, V.T. (2010) Relationship between flexibility and physical, chemical, and geometric characteristics of rotary nickel-titanium instruments. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics*, 110, 527–533.

Wycoff, R.C. & Berzins, D.W. (2012) An in vitro comparison of torsional stress properties of three different rotary nickel-titanium files with a similar cross-sectional design. *Journal of Endodontics*, 38, 1118–1120.

Zhou, H., Peng, B. & Zheng, Y. (2013) An overview of the mechanical properties of nickel–titanium endodontic instruments. *Endodontic Topics*, 29, 42–54.

Zupanc, J., Vahdat-Pajouh, N. & Schafer, E. (2018) New thermomechanically treated NiTi alloys – a review. *International Endodontic Journal*, 51, 1088–1103.

How to cite this article: Martins, J.N.R., Silva, E.J.N.L., Marques, D., Ajuz, N., Rito Pereira, M. & Pereira da Costa, R. et al. (2022) Characterization of the file-specific heat-treated ProTaper Ultimate rotary system. *International Endodontic Journal*, 00, 1–13. Available from: https://doi.org/10.1111/iej.13880

13652591, 0, Downloaded from https://onlinelibrary.wiley.com/doi/10.1111/iej.13880 by Universidade Nova De Lisboa, Wiley Online Library on [15/02/2023]. See the Terms and Conditions (https://onlinelibrary.wiley.com/terms-and-conditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License