Available online at www.giornaleitalianoendodonzia.it

ORIGINAL ARTICLE

Physico-mechanical properties of two different heat treated Nickel-Titanium instruments: in-vitro study

ABSTRACT

Aim: *This study aimed to define physico-mechanical properties of recently introduced thermally treated Edge Taper Platinum (ET Platinum) Nickel-Titanium (NiTi) instruments compared to conventional NiTi Edge Taper (ET) to disclose improvements obtained by heat-treatments.*
Methods: *ET and ET Platinum instruments (n=30/each) were rotated until fracture in a stainless-steel canal with 90°curvature angle and 5mm curvature radius. Time to fracture (TtF) and number of cycles to fracture (NCF) were calculated and results were analyzed using Mann-Whitney test (p=0.05). Fractured instruments underwent fractographic analysis under Field-Emission-Gun Scanning Electron Microscope (FEG-SEM). Superficial features of additional new ET and ET Platinum instruments were analyzed under SEM at increasing magnification with standardized angulations and positions. After inspection, ET and ET Platinum instruments were used in extracted teeth to shape 4 straight canals each to simulate clinical conditions and re-evaluated under SEM at the same points to detect superficial wear features. ET and ET Platinum brand new instruments were examined by differential scanning calorimetry (DSC) to assess transformation temperatures.*
Results: *Cyclic fatigue test showed a significant increase of fatigue resistance on ET Platinum compared to ET instruments (p=0.05). Fractographic analysis demonstrated a pattern of fatigue propagation on both instruments. SEM analysis on brand new instruments revealed a more regular surface of ET Platinum compared with ET and both instruments appeared with minimal alterations after the simulated clinical use. DSC analysis demonstrated higher amounts of martensite at 37°C in ET Platinum compared to ET instruments.*
Conclusions: *ET Platinum instruments displayed an improved mechanical behavior, possibly related to modified phase transformation temperatures induced by thermal treatment of the alloy during manufacturing process. These results suggest a safe use in clinical conditions.*

Riccardo Schiavon[1]
Gian Andrea Pelliccioni[1]*
Fausto Zamparini[1]
Giovanni Bolelli[2]
Andrea Spinelli[1]
Chiara Pirani[3]
Francesco Iacono[3]
Maria Giovanna Gandolfi[1]
Carlo Prati[1]
Luigi Generali[4]

[1]Endodontic Clinical Section, School of Dentistry, Department of Biomedical and Neuromotor Sciences DIBINEM, University of Bologna, Bologna, Italy.

[2]Department of Engineering Enzo Ferrari (DIEF), University of Modena and Reggio Emilia, Modena, Italy.

[3]Private Practice, Bologna, Italy

[4]Department of Surgery, Medicine, Dentistry and Morphological Sciences with Transplant Surgery, Oncology and Regenerative Medicine Relevance (CHIMOMO), University of Modena and Reggio Emilia, Modena, Italy.

Received 2023, September 19
Accepted 2023, October 20

KEYWORDS *Differential scanning calorimetry, cyclic fatigue, NiTi, endodontics, scanning electron microscope*

Corresponding Author
Gian Andrea Pelliccioni | Endodontic Clinical Section, Department of Biomedical and Neuromotor Sciences (DIBINEM), University of Bologna, Alma Mater Studiorum, Bologna | Italy.
Tel.: +39 051 2088126; fax: +39 051 225208 E-mail: gian.pelliccioni@unibo.it

Peer review under responsibility of Società Italiana di Endodonzia
10.32067/GIE.2023.37.01.28

Introduction

In the last decade many new designs, alloys, manufacturing processes, heat treatments and kinematics of Nickel-Titanium (NiTi) instruments have been introduced by several manufacturers (1). Recent strategies are focused to improve the mechanical properties of NiTi instruments through different thermomechanical treatments with the aim to optimize the alloy microstructure (2). In fact, heat-treatments are known to modify the intrinsic features of NiTi alloy due to the shift in crystalline structure (3, 4). The martensite/austenite transformation temperatures have been adjusted to allow a higher percentage of martensitic phase either at room, or body temperature, leading to more flexibile NiTi instruments and with an increased cyclic fatigue resistance compared to austenitic instruments; this is known to reduce intra-canal separation during clinical use (5).

Recently EdgeEndo (Albuquerque, NM, USA) has commercialized the heat-treated EdgeTaper Platinum (ET Platinum) rotary instruments. According to the manufacturer, ET Platinum instruments have the same design and geometric characteristic of EdgeTaper (ET), differing exclusively for the NiTi heat-treatment technology (EdgeTaper Platinum Brochure, EdgeEndo, Albuquerque, NM, USA).

ET are characterized by a bilobated triangular cross section with a progressive changing taper which tended to decrease at the middle and coronal portions. The operative system is based on an SX orifice opener (#19/12) an S1 (#17/09) and S2 (#20/06) series for shaping the canal and an F1 (#20/05), F2 (#25/05), F3 (#30/04), F4 (#40/05) and F5 (#50/05) series to finish the root canal instrumentation. The alloy of ET Platinum (Fire-Wire™) has been developed to improve flexibility and to enhance resistance (6). Fire-Wire™ is claimed to consist of an annealed heat-treated, cryogenically tempered NiTi alloy (7, 8) that should be in martensitic phase at body temperature.

The aim of the present study was to analyse the mechanical properties of recently introduced ET Platinum in comparison with not heat-treated ET instruments to better understand the improvements associated to the heat-treatment and if these instruments are suitable for the clinical use.

Materials and Methods

Cyclic Fatigue Resistance and Fractographic analysis

New ET (n=30, batch 070906170) and ET Platinum (n=30, batch 070918172) F1 size #20 with .06 variable taper and 25 mm length were rotated in a stainless steel (AISI 300) block containing a simulated canal with an angle of curvature of 90°, a radius of 5 mm and the centre of the curvature at 7 mm from the tip of the file (3) following manufacturer's recommendations (300 rpm) until fracture occurred. A fixed tool guaranteed a standardized placement of the instruments inside the artificial canal. Time to fracture (TtF) was recorded with a digital stopwatch and the numbers of cycles to fracture (NCF) was calculated using the following formula: $NCF = TtF(s) \times \text{rotation speed(rpm)} \times 60s^{1}$. Fragment length (FL) was measured using a digital caliper.

Fractured samples were cleaned to obtain a clear surface, in an ultrasonic bath containing a solution of isopropyl alcohol for 5 minutes at room temperature immediately before placing in the FEG-SEM. Instruments separated during cyclic fatigue test were subjected to fractographic analysis using a FEG-SEM (FEG-SEM - Nova NanoSEM 450, FEI, Eindhoven, Netherlands) and micrographs from 500x to 5000x were taken on the fractured surface.

SEM analysis and Wear test

Human extracted teeth were used to perform wear test. Theeth were removed due to orthodontic or periodontal reasons and were caries free without any root canal treatment. Patients were informed and agreed with the use for scientific research purpose. The approval of the Ethical Committee was obtained (Prot. n. 000083).

A Scanning Electron Microscope (SEM) (JSM-5200, JEOL, Tokyo, Japan) was used to evaluate superficial features of brand new additional instruments ET (n=3) and ET Platinum (n=3). Micrographs were taken at increasing magnification from 50× to 5000×, with standardized angulations and positions, at the tip of the instrument and on cutting edges at 5 mm from the tip. After the SEM examination these instruments were subjected to wear tests to simulate clinical conditions, using straight and round canals selected from a pool of extracted human teeth (9) evaluated radiographically with a mesio-distal and bucco-lingual angulation (4). Canal width of one-rooted teeth (n=24) was evaluated and measured on radiographs at 5 mm from the apex using a periodontal probe. Only teeth with canal width between 1 mm and 1,5 mm, at 5 mm far from the apex were accepted. Each tooth was sectioned at the cementum-enamel junction with a water-cooled diamond wheel saw in order to obtain a standard root length of 15 mm. Canal patency was verified with #10-15 stainless steel manual K-file and the working length was visually determined by insertion of a 25-mm #10 K file until its tip appeared at the apical foramen. Pre-flaring was achieved with an ET SX instrument 19/.12 19mm length (EdgeEndo, Albuquerque, New Mexico) and canal shaping was obtained with ET (n=3) and ET platinum (n=3) instruments 25 mm length S1 17/.06v and S2 20/.07v (EdgeEndo, Albuquerque, New Mexico). Instruments were used with a 16:1 reduction endodontic handpiece X-Smart Plus (Dentsply Maillefer, Baillagues, Switzerland) following manufacturer's recommendations at 300 rpm and 2.5 N·cm, with pecking motion. Irrigation was performed with 3 ml of 5% NaOCl (Niclor 5, Ogna, Muggiò, Italy) and 3 ml of 10% EDTA (Tubuliclean, Ogna, Muggiò, Italy). Every step was performed by the same expert operator. Each instrument was used in 4 canals, washed in an ultrasonic bath containing enzymatic solution for 10 min and then autoclaved at 134.8 °C (10). These multi-used instruments were observed at SEM after this wear test at the same points and angulations to identify the wear features. Wear was evaluated comparing the pre- and post-operative micrographs, to verify the presence of unwinding, microcracks, blade disruption and tip deformation (10-13).

Differential Scanning Calorimetry
Brand new ET (n=1) and ET Platinum (n=1) instruments were subjected to Differential Scanning Calorimetry (DSC, DSC2010, TA Instruments, New Castle, US-DE). Instruments were sectioned in their working portion in order to obtain a weight of 15mg and placed in an aluminum crucible to perform multiple heating and cooling cycles from -40 °C to 110 °C with a heating/cooling rate of 5 °C min^{-1}. Data were analyzed using software Universal Analysis 2000 (TA Instruments).

Statistical analysis
Data regarding the Cyclic Fatigue Resistance were analysed through Sigmaplot software (Systat, USA). Due to the non-normal distribution of the data (normality test>.05), Mann-Whitney test was performed (p=0.05) as statistical analysis.

Results

Cyclic Fatigue Resistance and Fractographic analysis
ET Platinum instruments exhibited significantly higher mean TtF and NCF compared to ET instruments (p<.05). The

Table 1

Descriptive analysis for NCF, time to fracture (TtF) expressed in seconds (s) and fragment length (FL) expressed in mm for EdgeTaper and EdgeTaper Platinum. Mean and standard deviations (SD) are represented.

	NCF, mean ± SD	TtF (s), mean ± SD	FL (mm), mean ± SD
EdgeTaper	139.52±41.11	27.9±3.9	7.71±1.33
EdgeTaper Platinum	319.89±187.36	63.98±12.9	8.39±1.88
P value	<0.05*	<0.05*	>0.05

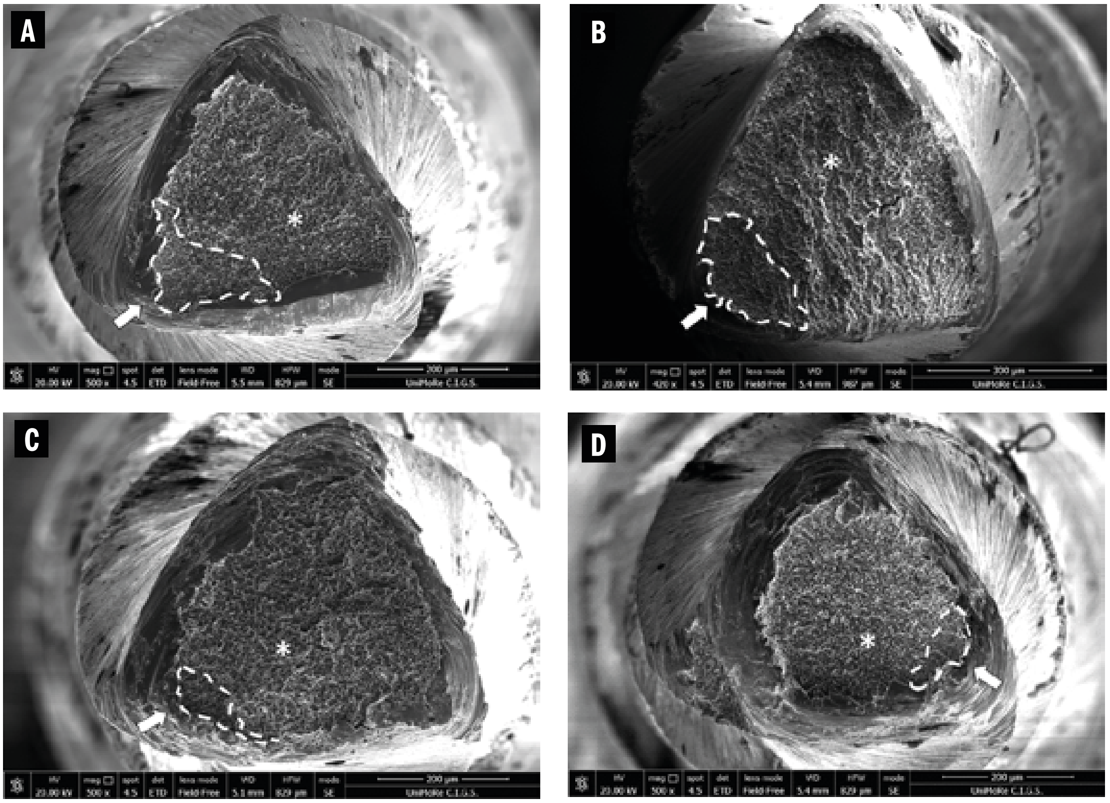


Figure 1
Fractographic analysis on ET (**A, B**) and on ET Platinum (**C, D**) instruments. In each micrograph the crack origin is identified and represented (white arrow), fatigue striation area (marked by white dotted line), and ductile fracture area (white asterisk) are noticeable.

difference in FL was not statistically significant (p>.05). Results are presented in Table 1.

On both groups of instruments, fractographic analysis revealed a small zone with fatigue striations next to a single crack origin in correspondence of an outer cutting edge, demonstrating a slow fatigue fracture propagation in this area. The remaining wider surface was covered by dimples, revealing the ductile fracture that caused the final rupture of the sample (Fig. 1). No pattern of torsional fracture was recognized in both instruments.

SEM analysis and Wear test

SEM analysis on brand new instruments revealed the presence of irregular grooves on the surface of ET, while ET Platinum exhibited a smooth regular surface (Fig. 2). Both instruments inspected as received from the manufacturer did not show fracture, microcracks, unwindings and the cutting edges were intact. Manufacturing residues were noticed on the surface of brand new ET instruments, but not on those of ET Platinum. After wear test, both types of instruments did not reveal surface degradation in terms of fracture, microcrack, unwinding, tip deformation and blade disruption. Both the groups of instruments showed the presence of residual dentinal debris on the cutting edges, which remained attached to the surface despite the ultrasonic bath and autoclaving cycle.

Differential Scanning Calorimetry

ET instruments exhibited a DSC peak that extends over a temperature range from +21.4 °C (Ms – martensitic start) to -14.9 °C (Mf – martensitic finish) during cooling (forward transformation) and a range from -7.6 °C (As – austenitic start) to +22.3 °C (Af – austenitic finish) during heating (reverse transformation) (Fig. 3). ET Platinum instruments showed a different behavior, with a DSC peak that extends over a temperature range from +33.5 °C (Ms – martensitic start) to +20.4

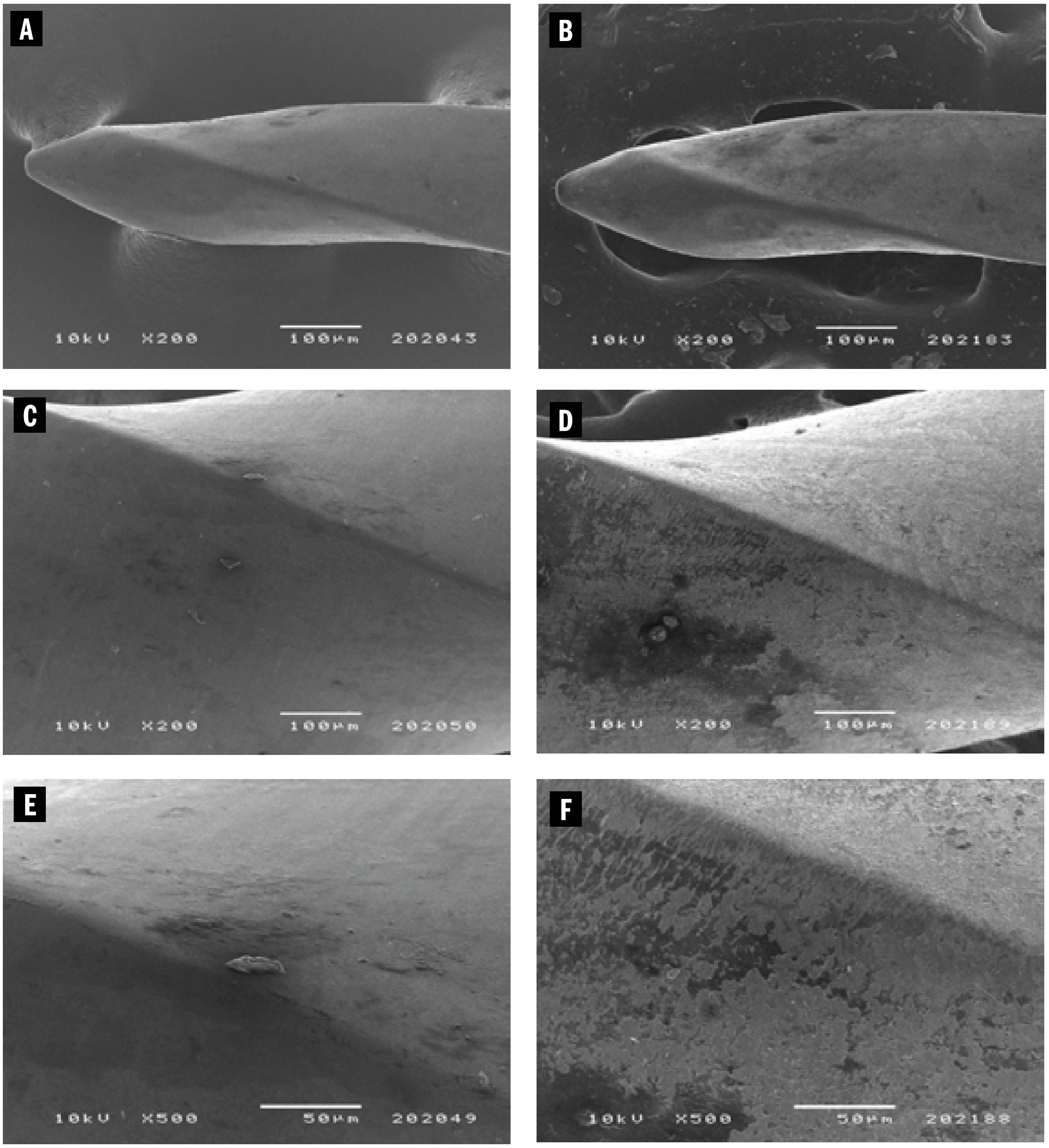


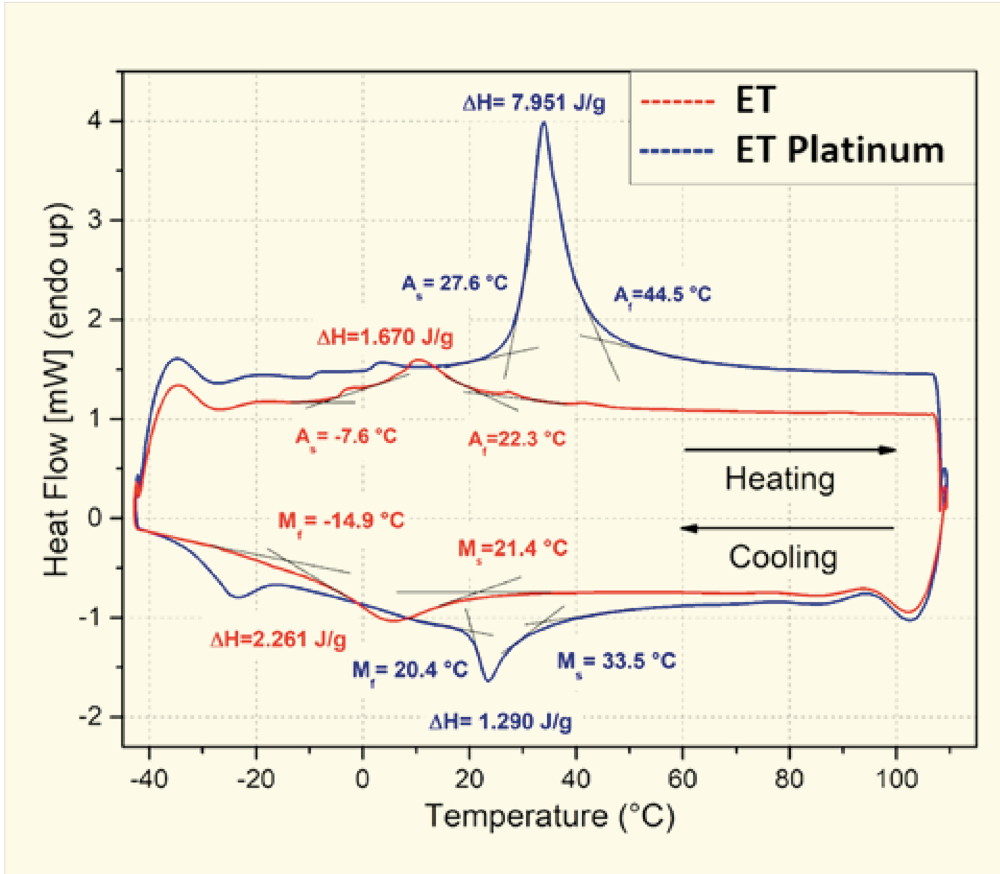
Figure 2
SEM analysis on ET Platinum before **(A, C, E)** and after **(B, D, F)** wear test. The outer surface exhibited a smooth regular texture. After wear test, no signs of surface degradation in terms of fracture, microcrack, unwinding, tip deformation and blade disruption were noticed. Residual dentinal debris on cutting edges after wear test, remained attached to the surface.

°C (Mf – martensitic finish) during cooling (forward transformation) and a range from +27.6 °C (As – austenitic start) to +44.5 °C (Af – austenitic finish) during heating (reverse transformation).

Discussion

This study pointed out that ET and ET Platinum rotary endodontic instruments share the same design, differing exclusively for the NiTi annealing heat-treatment. Considering this, every difference in their features may be attributed to this post-manufacturing process enabling comparability between the two groups. Therefore, these results could be translated also in other NiTi files which received the same heat treatment.
Results demonstrated that the heat-treatment greatly enhances the cyclic fatigue resistance because of the statistically significant increase in NCF of ET Platinum (heat-treated) compared to traditional ET (not heat-treated) instruments. In the present study the Fire-Wire™ alloy was found

Figure 3
Differential scanning calorimetry (DSC) graphic representing phase transformation temperatures (°C) of ET (red line) and ET Platinum (blue line). The "endo up" plotting convention was followed; whereby endothermic peaks are in the positive direction of the y-axis and vice versa. The temperatures associated with the onset and completion of the austenite → martensite transformation (direct transformation) during cooling, labelled as M_s and M_f, and of the the martensite → austenite transformation during heating (reverse transformation), respectively labelled A_s and A_f, are indicated on the plot. Transformation enthalpies (ΔH) associated with each peak were calculated as the ratio of the overall amount of heat absorbed/released (respectively) by the sample during the transformation and the sample mass, and are specified on the plot next to the corresponding peaks, in units of J/g.



to be more resistant to cyclic fatigue when compared with the conventional NiTi alloy. As a matter of fact, the NCF of ET Platinum was almost 2.3 times higher compared to the austenitic ET, suggesting a safer clinical use especially in curved canals. The literature provides a comparison between the cyclic fatigue resistance of ET Platinum and another instrument of similar characteristics: the former still appears to be superior tested in a 60° 5 mm radius curved artificial canal (6). Similarly, in previous studies (8) Fire-Wire™ alloy instruments outlasted instruments made by M-Wire and Gold wire alloys both in continuous rotation and reciprocation. Scott et al. (2019) also reported a slightly different design of the tested instruments as a reason for the difference in cyclic fatigue resistance in their study (8). A comparison between ET Platinum and ProTaper Gold (14) reported a statistically significant higher cyclic fatigue resistance of ET Platinum, despite a lesser resistance to torsional load, thus confirming the results of Alcalde et al. (15). As mentioned, in the present study the tested instruments differed only for the heat treatment of the alloy and this eliminates the variables that can be connected with a different morphology of the compared instruments, such as section area, section design and taper. The homogeneity of the instrument's morphology between the two groups, and the rigorous set-up of the experimental conditions can explain why there was not statistically significant difference in the FL between the two groups.

FEG-SEM analysis of fractured instruments showed the typical pattern of cyclic fatigue fracture (16, 17), validating the chosen cyclic fatigue test procedure and confirming that no torsional load was simultaneously exerted on the instruments. In fact, SEM analysis of the fractured surface revealed a single crack origin on the cutting edge of both instruments, with a relatively small propagation area showing the typical fatigue striations, while the remaining part was covered by dimples and showed a ductile fracture pattern; Piao et al. (2014) conducted a similar and more extensive fractographic analysis procedure with different instruments (18). Heat-treated ET Platinum appears to have larger fatigue propagation area compared to ET, suggesting a longer crack propagation stage before the final fracture, compatible with the higher duration obtained through the cyclic fatigue test. Moreover, ET Platinum instruments presented more regular fractured margins, compared to the traditional ET. This could suggest that traditional ET had a sudden ductile fracture rather than a progressive propagation of the crack (17).

SEM analysis was selected for the assessment of the morphological characteristics of the instruments before and after wear testing, to verify the onset of superficial defects (3, 10, 11, 12, 19). Both ET and ET Platinum passed intact the wear test, which simulates clinical conditions, with no detectable alterations of their outer morphology. Both types of instruments did not reveal any surface degradation in the form of microcracks, tip deformation, fracture, unwinding, blade disruption during the test; so, it can be assumed that both instruments have high wear resistance under clinical conditions. Confirming this, Alfawaz et al. (2022) found ET Platinum to be less affected by working in a solution of NaOCl compared to other instruments (20). Interestingly, ET instruments revealed the presence of many irregular grooves on the surface before the test, unlike ET Platinum that exhibited a smoother and more regular surface. Nevertheless, the finding of an irregular surface on some new instruments, probably ascribed to some differences during the manufacturing process, did not affect the wear resistance (i.e. comparable results between groups). On the contrary, it can be speculated that the presence of an irregular surface can affect the cyclic fatigue resistance, contributing to the in significantly different results between ET and ET Platinum.

DSC assessment of martensitic conversion temperatures can help in predicting the behavior of NiTi rotaries in different environments (21-24). In the present evaluation DSC analysis revealed the phase transformation points of martensite and austenite in EdgeEndo Fire-Wire™ alloy. Because the reverse transformation peak of the ET Platinum file is centered approximately around the body temperature (37 °C), it is inferred that this file has a higher amount of martensite at the body temperature compared to ET, which is inferred to be completely austenitic, confirming the findings of a recent study (25). Thermo-mechanically treated NiTi alloys have been widely reported to possess an increased flexibility and an augmented cyclic fatigue resistance (5) and DSC data of the present study suggests that ET Platinum benefits from a higher amount of martensite, which is expected to make the material more flexible, hence more resistant to rotating bending fatigue. Considering the in-vitro design of the study, it can be completed with investigations to evaluate the impact of Fire-Wire™ alloy on the actual clinical effectiveness of the instruments.

Conclusions

Heat-treated NiTi ET Platinum instruments revealed an improved mechanical behavior compared to traditional NiTi ET, possibly related to a modified phase transformation temperature of the wire induced by thermal treatment during manufacturing process. Fire-Wire features analyzed in the present study suggest a safe use in clinical conditions.

Clinical Relevance

Clinically, the enhanced fatigue resistance and improved surface characteristics of ET Platinum instruments suggest a safer and more effective use in challenging endodontic procedures, especially in curved canals. The heat treatment process employed in ET Platinum instruments leads to a more regular surface and higher resistance to cyclic fatigue, which could significantly reduce the risk of instrument fracture during clinical procedures.

Conflicts of Interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Acknowledgements

Funding
Master in Clinical Endodontics – DIBINEM – Alma Mater Studiorum University of Bologna was determinant for the funding of most of this research project. The Authors report that part of the research was funded from the FAR project (University of Modena and Reggio Emilia Funds for Research) entitled Microstructural and Mechanical investigation of Nickel-Titanium Endodontic instruments – MMiNTEndo.

Institutional Review Board Statement
The study was conducted in accordance with the Declaration of Helsinki, and approved by the the Ethical Committee (Prot. n. 000083) "Comitato Etico di Area Vasta Emilia Centro della Regione Emilia-Romagna located in Azienda Ospedaliero - Universitaria di Bologna, Policlinico S.Orsola-MalpighiVia Albertoni, 15 - 40138 Bologna".

References

1 Peters OA, Bahia MG, Pereira ES. Contemporary root canal preparation: innovations in biomechanics. Dent Clin North Am 2017; 61: 1193-7
2 Shen Y, Zhou H, Zheng Y, Peng B, Haapasalo M. Current challenges and concepts of the thermomechanical treatment of nickel-titanium instruments. J Endod 2013; 39:163-172.
3 Pirani C, Iacono F, Generali L, Sassatelli P, Nucci C, Lusvarghi L, Gandolfi MG, Prati C. HyFlex EDM: superficial features, metallurgical analysis and fatigue resistance of innovative electro discharge machined NiTi rotary instruments. Int Endod J. 2016;49:483-93.
4 Generali L, Puddu P, Borghi A, Brancolini S, Lusvarghi L, Bolelli G, Consolo U, Pedullà E. Mechanical properties and metallurgical features of new and ex vivo used Reciproc Blue and Reciproc. Int Endod J 2020; 53:250-264.
5 Zupanc J, Vahdat-Pajouh N, Schafer E. New thermomechanically treated NiTi alloys - a review. Int Endod J 2018; 51: 1088-103.
6 Jamleh A, Alghaihab A, Alfadley A, Alfawaz H, Alqedairi A, Alfouzan K. Cyclic Fatigue and Torsional Failure of EdgeTaper Platinum Endodontic Files at Simulated Body Temperature. J Endod. 2019;45:611-614
7 Khalil W, Natto Z. Cyclic fatigue,bending resistance, and surface roughness of ProTaper Gold and EdgeEvolve files in canals with single- and double curvature. Restor Dent Endod 2019; 44: 1–9.
8 Scott R, Arias A, Macorra JC, Govindjee S, Peters OA. Resistance to cyclic fatigue of reciprocating instruments determined at body temperature and phase transformation analysis. Aust Endod J. 2019 Dec;45:400-406.
9 Kosti E, Zinelis S, Molyvdas I, Lambrianidis T. Effect of root canal curvature on the failure incidence of profile rotary Ni—Ti endodontic instruments. Int Endod J 2011;44:917-25.
10 Pirani C, Paolucci A, Ruggeri O et al. Wear and metallographic analysis of Wave One and Reciproc NiTi instruments before and after 3 uses in root canals. Scanning 2014; 36, 517–25.
11 Troian CH, So´ MVR, Figueiredo JAP, Oliveira EPM. Deformation and fracture of RaCe and K3 endodontic instruments according to the number of uses. Int Endod J 2006; 39, 616–25.
12 Caballero H, Rivera F, Salas H. Scanning electron microscopy of superficial defects in Twisted files and Reciproc nickel-titanium files after use in extracted molars. Int Endod J 2015; 48, 229–35.
13 Azizi, A., Prati, C., Schiavon, R., Fitzgibbon, R. M., Pirani, C., Iacono, F., Pelliccioni, G. A., Spinelli, A., Zamparini, F., Puddu, P., Bolelli, G., & Generali, L. (2021). In-Depth Metallurgical and Microstructural Analysis of Oneshape and Heat Treated Onecurve Instruments. European Endodontic Journal, 6, 90–97. https://doi.org/10.14744/eej.2021.63634
14 Zanza, A., Russo, P., Di Matteo, P., Reda, R., Di Nardo, D., Gambarini, G., & Testarelli, L. (2022). Mechanical properties and metallurgical features of two similar endodontic rotary instruments with different heat treatments (FireWire™ and Gold). Science progress, 105(2), 368504221103763.
15 Alcalde, M., Duarte, M., Amoroso Silva, P. A., Souza Calefi, P. H., Silva, E., Duque, J., & Vivan, R. (2020). Mechanical Properties of ProTaper Gold, EdgeTaper Platinum, Flex Gold and Pro-T Rotary Systems. European endodontic journal, 5(3), 205–211.
16 Hull D: Fractography: observing, measuring and interpreting fracture surface topography, 3rd ed. Cambridge University Press, 1999.
17 Brooks CR, Choudhury A. Failure Analysis of Engineering Materials, 2nd ed. New York, NY, USA: McGraw-Hill; 2002.
18 Piao J, Miyara K, Ebihara A, Nomura N, Hanawa T,

Suda H: Correlation between cyclic fatigue and the bending properties of nickel titanium endodontic instruments. Dent Mater J, 33:539-44, 2014.

19 Eggert C, Peters O, Barbakow F. Wear of nickel– titanium lightspeed instruments evaluated by scanning electron microscopy. J Endod 1999; 25, 494–7.

20 Alfawaz H, Alqedairi A, Alhamdan M, Alkhzim N, Alfarraj S, Jamleh A. Effect of NaOCl and EDTA irrigating solutions on the cyclic fatigue resistance of EdgeTaper Platinum instruments. BMC Oral Health. 2022 May 22;22(1):195.

21 Alapati SB, Brantley WA, Iijima M, et al. Metallurgical characterization of a new nickel-titanium wire for rotary endodontic instruments. J Endod 2009a;35:1589–93.

22 Alapati SB, Brantley WA, Iijima M, et al. Micro-XRD and temperature-modulated DSC investigation of nickel-titanium rotary endodontic instruments. Dent Mater 2009b;25:1221–9.

23 Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH. Differential scanning calorimetric studies of nickel-titanium rotary endodontic instruments. J Endod 2002a;28:567–72.

24 Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH. Differential scanning calorimetric studies of nickel-titanium rotary endodontic instruments after simulated clinical use. J Endod 2002b;28:774–8.

24 Martins JNR, Silva EJNL, Marques D, Pereira MR, Ginjeira A, Silva RJC, Braz Fernandes FM, Versiani MA. Mechanical Performance and Metallurgical Features of ProTaper Universal and 6 Replicalike Systems. J Endod. 2020;46:1884-1893.