coatings

Article

Comprehensive Characterization of Blue Wire NiTi File Failure: A Comparative Analysis of Cyclic Fatigue and Torsional Resistance Properties

Reem M. Barakat [1], Rahaf A. Almohareb [2,*], Fahda N. Algahtani [2], Amal A. Altamimi [2], Jenan I. Alfuraih [2], Lena S. Bahlol [2] and Ahmed Jamleh [3]

1 Dental Clinics Department, King Abdullah bin Abdulaziz University Hospital, Princess Nourah Bint Abdulrahman University, P.O. Box 84428, Riyadh 11671, Saudi Arabia; rmbarakat@pnu.edu.sa

2 Department of Clinical Dental Sciences, College of Dentistry, Princess Nourah Bint Abdulrahman University, P.O. Box 84428, Riyadh 1167, Saudi Arabia; fnalgahtani@pnu.edu.sa (F.N.A.); amaltamimi@gmail.com (A.A.A.); lenabahlol@gmail.com (L.S.B.)

3 Department of Restorative Dentistry, College of Dental Medicine, University of Sharjah, Sharjah 27272, United Arab Emirates; aojamleh@gmail.com

* Correspondence: raalmohareb@pnu.edu.sa

Abstract: This study compared the fatigue resistance and elemental composition of two blue heat-treated nickel–titanium (NiTi) files used in root canal preparation as follows: Tia Tornado Blue (TTB) and Race Evo (RE) file systems. For cyclic fatigue testing, the two systems were tested where each file was rotated inside an artificial metal canal submerged in either sodium hypochlorite or saline solution until fracture. Time to fracture was recorded. For torsional fatigue testing, the file tip was secured while the file was allowed to rotate at a fixed rate until fracture. Torque at failure was recorded. The two experiments were performed at simulated body temperature and the length of fractured segments was measured. Statistical analysis was carried out with a significance level (p-value) set at 5%. The mean cycles to fracture for RE were superior to that of TTB irrespective of the solution used ($p < 0.05$). TTB's cyclic fatigue resistance decreased in NaOCl ($p < 0.0001$). RE demonstrated lower torque at failure ($p = 0.002$). All files were fractured at comparable lengths ($p = 0.218$). Although RE is considered more resistant to cyclic fatigue, it showed inferior torsional resistance compared with TTB. The NaOCl negatively affected the TTB's cyclic fatigue resistance.

Keywords: blue wire; corrosion; cyclic fatigue; nickel titanium; sodium hypochlorite; torsional resistance

Citation: Barakat, R.M.; Almohareb, R.A.; Algahtani, F.N.; Altamimi, A.A.; Alfuraih, J.I.; Bahlol, L.S.; Jamleh, A. Comprehensive Characterization of Blue Wire NiTi File Failure: A Comparative Analysis of Cyclic Fatigue and Torsional Resistance Properties. *Coatings* **2024**, *14*, 361. https://doi.org/10.3390/coatings14030361

Academic Editors: Monika Lukomska-Szymanska and Louis Hardan

Received: 26 February 2024
Revised: 12 March 2024
Accepted: 16 March 2024
Published: 19 March 2024

1. Introduction

Quality root canal preparation is essential to achieving a successful root canal treatment [1]. This involves shaping and disinfecting the root canal with file instruments and irrigating solutions, respectively. In the past, only stainless-steel files were available to shape root canals [2]. This limited the ability of the clinician to safely shape complex curvatures without creating deviations, ledges, and blockages that compromise treatment success. Nickel–titanium (NiTi) files have heralded a new era in root canal treatment. They allow for faster and more predictable preparation of the canal system. This is made possible by virtue of the unique metallurgical properties of the NiTi alloy, such as super-elasticity and shape memory [2]. However, unexpected NiTi file fractures due to internal stress that might take place during canal preparation are a significant concern. There are two main fracture scenarios. The first scenario is cyclic fatigue, which is created by the repeated compressive and tensile stresses caused while rotating the file inside a curved canal. The second one is torsional fatigue, where the file tip gets stuck in the canal while the shank keeps rotating [3]. Both fatigue mechanisms induce stresses, which, in turn, reduce the file hardness and modulus of elasticity, as evidenced by nanoindentation studies [4,5].

The first generation of NiTi files, termed conventional NiTi, exhibits an austenite structure at 37 °C, as their austenite finish temperature is below body temperature. The austenite crystal lattice transforms into martensite under the stress induced by inserting the file into the curved root canal [6]. Successively, manufacturers have employed new manufacturing processes and different raw alloy compositions to improve the file's resistance and performance. By altering the temperatures at which the crystalline-phase transformation occurs, they were able to obtain NiTi alloys with more favorable mechanical properties at body temperature, thereby improving the reliability of NiTi files. Through heat treatment, the NiTi finish temperature for austenite transformation can be elevated. This alteration leads to an alloy characterized by a higher proportion of martensitic and R-phase components at body temperature [7,8]. Consequently, these files demonstrate enhanced flexibility and fatigue resistance [9–11].

Certain manufacturers have developed a thermos-mechanical manufacturing process that creates blue NiTi. Its distinct blue color is due to a protective oxide surface layer that is retained after machining and thermal treatment. These files feature a higher proportion of stable martensite, resulting in a more flexible NiTi alloy [12]. This enhancement has led to improved performance compared with conventional NiTi files [13,14]. Recently, two new blue NiTi file systems have been introduced. Race Evo (RE) (FKG Dentaire SA, La Chaux de Fonds, Switzerland) is designed to be operated at a relatively high rotation speed (800 rpm). It has a triangular cross-section with alternating cutting edges that prevent file threading into the canal. These files have shown improved cyclic fatigue resistance that is not affected by autoclave sterilization [15,16]. Tia Tornado Blue (TTB) (Tiadent, Houston, TX, USA) is another novel blue rotary file system made with heat-treated technology. While the manufacturers claim that it has superior torque strength and resistance to cyclic fatigue [17], a previous study showed no improvement in cyclic fatigue resistance in comparison with other blue NiTi files [18]. It is essential to partner mechanical root canal preparation with chemical irrigation to help lubricate files, flush out debris, and most crucially, achieve the disinfection of the root canal system and dissolve pulp tissues without damaging periapical tissues [1]. To date, sodium hypochlorite (NaOCl) remains the intracanal irrigant of choice in endodontics [19]. It is employed in concentrations ranging from 0.5% to 5.25% as an affordable antibacterial agent and lubricant. NaOCl works by breaking proteins into amino acids, while free chlorine dissolves both living and dead tissues [20]. Although NiTi files exhibit excellent resistance to corrosion, they have been prone to corrosive defects when immersed in NaOCl [21,22]. The corrosive sites are suspected of acting as stress accumulation points that lead to crack propagation, jeopardizing the NiTi files' mechanical properties [22,23]. Previous studies evaluated the effects of NaOCl on the NiTi files and reported that NaOCl at specific concentrations and temperatures influenced the cyclic fatigue resistance of certain NiTi files [24–26].

Limited articles are available on the cyclic fatigue resistance of blue NiTi files [18,27]. However, no studies have examined both cyclic fatigue and torsional resistance of blue NiTi files at body temperature, let alone their performance under conditions simulating clinical situations such as irrigation with NaOCl. Understanding blue NiTi file properties can influence dentists' decisions in the clinic and shed light on their effectiveness in complex root canal systems, especially given the absence of a standardized benchmark for NiTi instrument quality before market introduction [28]. Therefore, the purpose of this study was to compare the cyclic fatigue and torsional resistance of RE and TTB files under simulated clinical conditions and explore their elemental composition. The null hypothesis was that there is no difference between the tested NiTi file systems in terms of cyclic fatigue and torsional failure.

2. Materials and Methods

Sample size calculation was performed using G*Power software version 3.1.9.6 (Heinrich-Heine-Universität, Düsseldorf, Germany), estimating the power at 0.90 and a probability of falsely rejecting the null hypothesis Type 1 error $\alpha = 0.05$. The estimated sample size was 15 in each group. A total of ninety brand-new files were used in this study (45 TTB and 45 RE files). All files had a tip size of 30 (0.30 mm) with a 0.04 mm constant taper and a 25 mm length. The files were inspected for flaws or defects under 13.6× magnification (A6 series Global surgical corporation; Saint Louis, MO, USA) without any need for exclusion. The files were randomly divided into four equal groups (n = 15 per group) based on the NiTi file and the type of irrigating solution used.

2.1. Cyclic Fatigue Testing

Thirty files from each NiTi system were used. Each file was inserted into an artificial canal that was milled in a stainless-steel block in such a way that 19 mm of the file from its tip was inside the canal [29]. The artificial canal had a 60° angle of curvature with a 5 mm radius. Its internal dimensions were larger than the NiTi files by 0.1 mm. The block was stabilized in a container filled with either 5.25% NaOCl or saline solution. A glass slab was placed above the artificial canal to prevent spillage and keep track of the file's rotations until it fractured. To simulate intracanal temperature, the solution's temperature was set at 35 ± 1 °C assessed by a digital thermometer. The files were operated using an electric endodontic motor (X-Smart Plus; Dentsply Sirona; Charlotte, NC, USA) according to the manufacturers' recommendations regarding speed and torque. After the file was inserted into the metal canal, the motor was operated at a speed of 350 rotations per minute (rpm) with 2 N·cm torque for TTB files and 800 rpm with 1.5 N·cm torque for RE until fracture occurred (Figure 1). The time to fracture, audibly or visually recognized, was recorded, and subsequently, the number of cycles to fracture was calculated. Furthermore, the length of the fractured segment was measured carefully using a digital caliper.

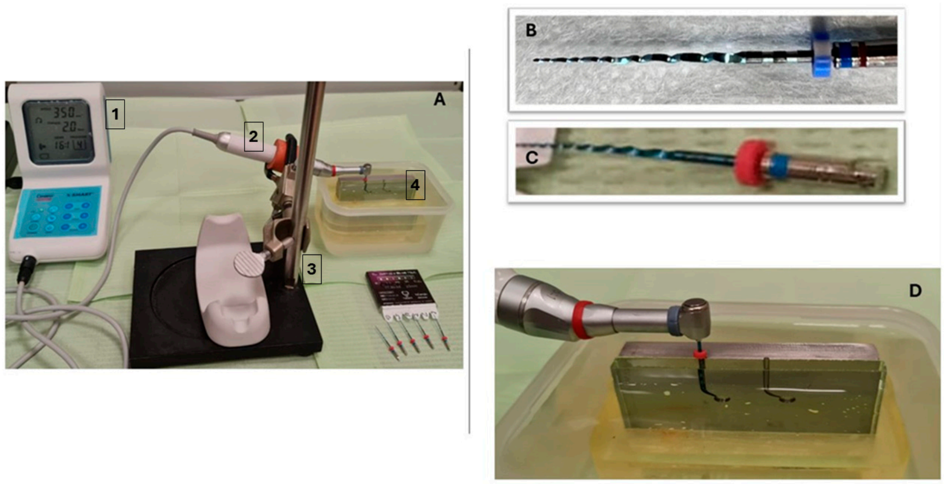

Figure 1. (**A**) Photo of the experimental set-up for testing the NiTi files' cyclic fatigue showing: (1) the Endodontic Electric motor; (2) the handpiece fixed on a stand (3); and (4) the stainless-steel block with artificial canal. (**B**) RE files and (**C**) TTB files. (**D**) Files placed in artificial canals immersed in the irrigant.

2.2. Torsional Resistance Test

Fifteen files from each NiTi system were used. The apical part (5 mm) of the file was firmly secured using a stainless-steel pen vise. A large vise was used to mount the X-Smart Plus handpiece so that it could keep the file upright. The pen vise, along with the file, was securely mounted on the top of a torque gauge device (TT01; Mark-10 Corporation, Long Island, NY, USA). The file was rotated clockwise at a speed of 40 rpm until a fracture occurred by applying uniform torsional stress. The experiment was conducted after zeroing the gauge at a simulated intracanal temperature of 35 ± 1 °C as confirmed by a thermocouple. The torque-at-failure values were noted. The length of the fractured segment was also measured carefully.

2.3. Scanning Electron Microscope

Two fractured files from each group in the two tests were cleaned with absolute alcohol in an ultrasonic bath. The topographical characteristics of the fractured surfaces were then examined using a scanning electron microscope (SEM; JSM-7001F, JEOL, Tokyo, Japan). Photomicrographs of the fractured surfaces were taken at ×230 and ×1000 magnifications.

2.4. Energy-Dispersive X-ray Spectroscopy Analysis (EDX)

Three RE and TTB underwent EDX analysis using a JSM-IT500HR EDX detector (STD-PC80, JEOL, Tokyo, Japan). The following exposure parameters were used: acceleration voltage of 15 kV at a magnification of ×1000. The samples were coated with platinum using an auto-fine coater (JEC-3000FC, Jeol, Japan) for 80 s at 10 µA. The elemental composition of the files was analyzed, and the atomic weight percentages were measured at three different locations on the file.

2.5. Statistical Analysis

The data were analyzed using SPSS software version 22 (SPSS Inc., Chicago, IL, USA) at a 5% significance level. The data were checked using the Shapiro–Wilk test. The cyclic fatigue results were not normally distributed. Thus, the Kruskal–Wallis test followed by the post hoc Mann–Whitney test was conducted. For the torsional resistance and the length of separated fragments, independent *t*-test and one-way ANOVA were used.

3. Results

Table 1 shows the cyclic fatigue results for the tested NiTi systems in both solutions. RE files showed superior resistance with an increased number of cycles to fracture compared with TTB, irrespective of the solution type ($p < 0.0001$). However, TTB's resistance to cyclic fatigue was negatively influenced by immersion in NaOCl ($p < 0.0001$), unlike RE ($p = 0.09$). The length of fractured segments due to cyclic fatigue was found to be comparable in all groups, ranging from 4.29 to 4.62 mm ($p = 0.30$).

Table 1. Mean and standard deviation of the number of cycles to fracture and length of separated fragments for both TTB and RE files rotating in different irrigating solutions.

NiTi System	Solution Media	Cycles to Fracture	Fragment Length (mm)
Tiadent Tornado Blue (TTB)	Normal saline (n = 15) NaOCl (n = 15)	3514 ± 740.31 [a] 2564.72 ± 421.15 [b]	4.29 ± 0.69 4.54 ± 0.52
Race Evo (RE)	Normal saline (n = 15) NaOCl (n = 15)	6651.56 ± 1402.45 [c] 7584 ± 1120.0 [c]	4.56 ± 0.34 4.62 ± 0.38
p-value		0.00	0.30

[a, b, c] Different lower-case letters represent statistically significant differences between groups.

In terms of torsional resistance, RE demonstrated significantly lower torque at failure (1.03 ± 0.10 N·cm) compared with TTB (1.21 ± 0.14 N·cm) ($p = 0.002$). However, there was no difference in the length of fractured segments, ranging from 5.05 and 5.15 mm ($p = 0.218$). The complete data set for the cyclic and torsional fatigue tests can be found in Tables S1 and S2 as a Supplementary Material.

SEM photomicrographs of fractured surfaces due to cyclic fatigue and torsional failure can be seen in Figures 2 and 3, respectively.

The EDX analysis revealed variations in the atomic weight percentages of nickel (Ni) and titanium (Ti) between TTB and RE files. Furthermore, aluminum (Al) and zirconium (Zr) were detected exclusively in RE files. Silicone (Si), on the other hand, was detected in both TTB and RE files (Table 2) (Figure 4).

Table 2. The mean atomic weight percentages (%) of elements revealed by the EDX analysis of TTB and RE files.

Spectrum	Ni	Ti	O	C	Si	Al	Zr
TTB	36.82	37.64	24.76667	16.91	0.75	-	-
RE	27.66714	29.31286	25.55429	20.554	0.90	0.88	0.36

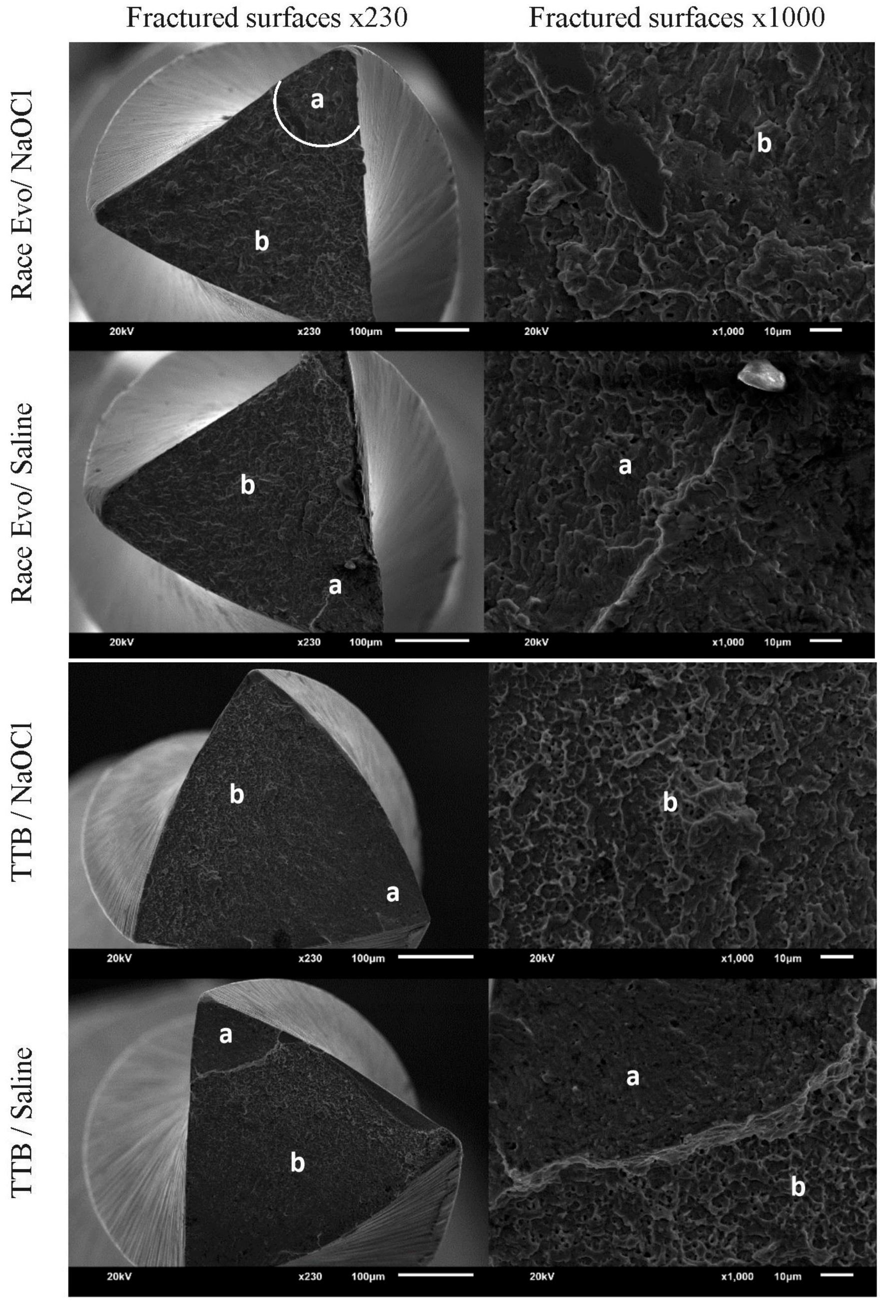

Figure 2. Scanning electron microscope photomicrographs of fractured surfaces of TTB and RE after cyclic fatigue resistance testing in NaOCl and saline at ×230 and ×1000 magnification. (a) shows an area of fatigue linear striations, while (b) presents dimpling and micro-voids characteristic of ductile fracture.

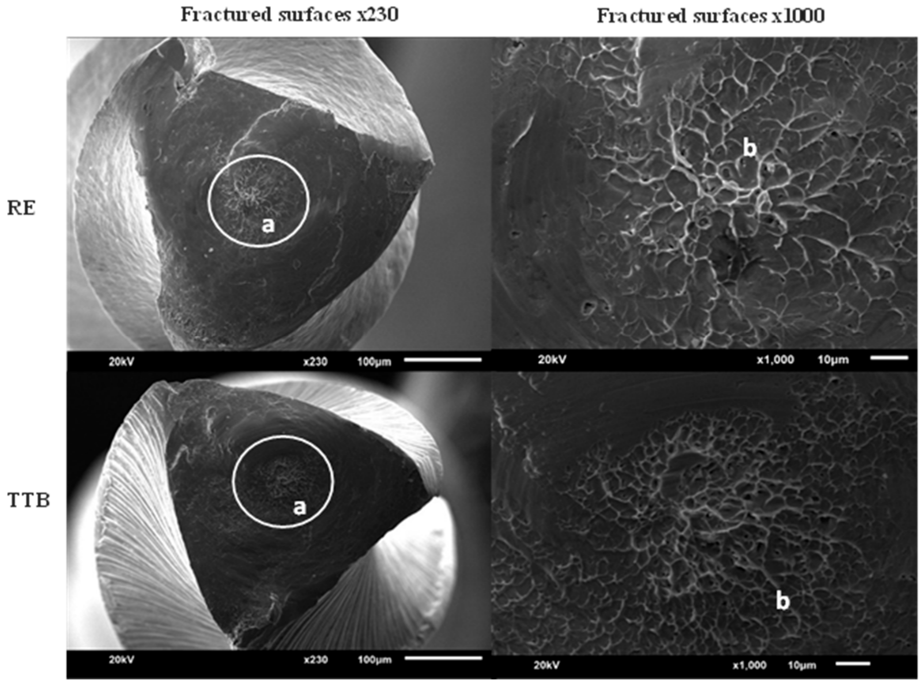

Figure 3. Scanning electron microscope photomicrographs showing TTB and RE fractured surface after torsional resistance testing at $\times$230 and $\times$1000 magnification. (a) shows the central area of the shear-induced concentric mark, while (b) presents a dimpling characteristic of brittle fracture.

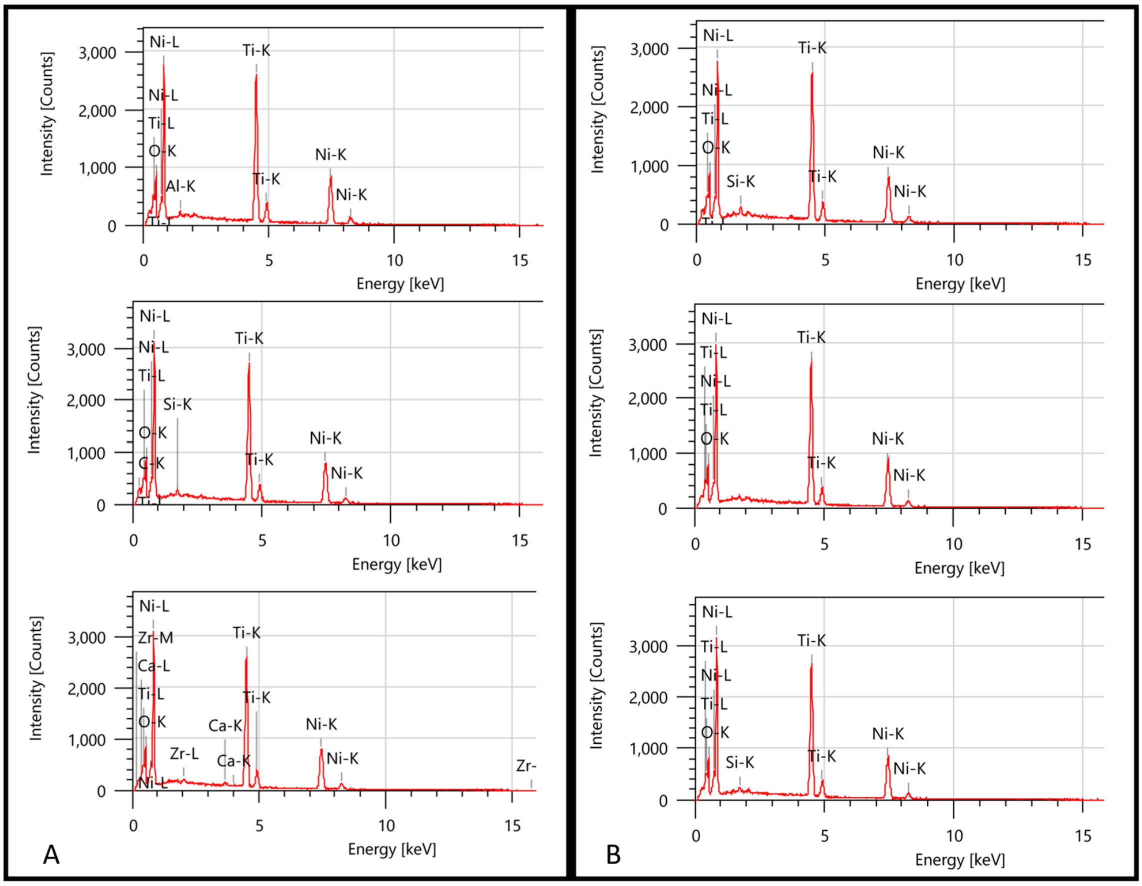

Figure 4. EDX analysis of Race Evo files (**A**) and Tiadent Tornado Blue files (**B**) in different locations through the length of the file.

4. Discussion

In this study, two blue NiTi file systems were evaluated in terms of cyclic fatigue and torsional resistance at simulated body temperatures. Compared with TTB, RE files showed superior resistance to cyclic fatigue but lower resistance to torsional stress. Thus, the null hypothesis was rejected.

Although the tested systems have similar dimensions and cross-sections, the RE file has a lower metal mass volume due to its alternating file blade design. It is known that the file's cross-section has an influence on cyclic fatigue resistance [30]. Furthermore, previous studies compared RE with the conventional Race file and reported that the blue technology enhanced the cyclic fatigue resistance of RE [15,16]. On the other hand, a study comparing RE to the R-motion reciprocating file found that the latter required more time to fracture, possibly due to motion kinematics [16]. The blue technology in RE allows for phase transition from martensite to austenite to occur at temperatures close to intracanal temperature (28 to 35 °C) [16]. Moreover, the chemical composition of the employed NiTi wire and the heat manufacturing procedures they undergo might cause these differences, which are also seen in other heat-treated NiTi files with identical geometric designs, such as ProTaper Gold and EdgeTaper Platinum [11,31].

The cyclic fatigue resistance was tested in two solutions (saline or NaOCl). Although the file was in short contact with the solution, there was a significant reduction in the number of cycles to fracture only TTB in NaOCl. NaOCl is known to develop corrosive zones on the surface [2]. RE was not affected due to the presence of the blue titanium oxide layer, which is known to improve resistance to wear and decrease surface roughness with autoclave sterilization [13,15,32]. The negative effect of NaOCl on cyclic fatigue resistance was noticed in some NiTi systems, such as ProTaper Gold, EdgeTaper Platinum, and D-Race [2,33,34], but not in others (Wave One, Wave One Gold, and HyFlex EDM) [26,35,36]. The contradictory results in these studies may be attributed to differences in the methodology and the alloy properties, design, and kinematics of the tested files. The methodology used in this study places the tested files in an artificial canal filled with the irrigating solution for a short duration. More investigations are warranted to study the surface properties of heat-treated files such as RE and their performance under different solutions.

Although RE improved cyclic fatigue, it was unable to show better torsional resistance than TTB. This is in accordance with several previous findings where files with superior resistance to cyclic fatigue were found to have less resistance to torsional stresses due to their heat treatment [37,38]. However, both files in this study were heat-treated. So, the results could be related to the file design and chemical composition. Torsional resistance increases with decreasing pitch (increasing number of threads) as well as a larger cross-sectional area [31,39]. However, the two NiTi files used in the study had a similar tip size, taper, and triangular cross-section design. Unlike TTB, RE carries the Race unique alternating cutting-edge design, which means fewer threads to reduce the screwing effect and allow for better control of the file's progression in the canal [40]. Although the mean torque required to fracture RE in this study (1.03 N·cm) is less than that reported by a previous study (1.26 N·cm) [16], this may be due to different methodologies in running torsional testing. Both studies reported a value lower than what has been recommended by the manufacturer (1.5 N·cm). The same applies to TTB.

The files fractured at relatively similar points (4–5 mm) from the tip, which is close to the point of maximum flexure or torsion. This shows that all files were subjected to maximum stress points in that location, which is in accordance with previous studies [2,25].

The EDX analysis of the files revealed a substantial presence of alloying elements that affect phase transformation temperature. This observation affirms a more martensitic nature associated with increased flexibility and resistance to fracture. Several of these alloying elements, such as oxygen (O), carbon (C), and aluminum (Al), have been documented in prior studies on other shape memory NiTi files [41,42].

Interestingly, silicon (Si) was identified in both files. Si acts as an alloying element that elevates the phase transformation temperature, contributing to martensite phase stability at room temperature and providing a cost-effective alternative to more expensive alloying materials [43,44].

Additionally, the RE files exhibited the presence of zirconium (Zr). Previous studies have indicated that NiTi surface treatments involving Zr can result in smoother surfaces and improved resistance to metal fatigue [45,46]. The presence of Al and Zr in RE files may also explain why their cyclic fatigue resistance remained unaffected by NaOCl [47].

Although the testing methods used in this study provided reliable, standardized conditions that allow for different mechanical properties to be tested, their main limitation is that they do not fully replicate the clinical situation. The cyclic fatigue and torsional testing used lacked the dynamic movement the file is subjected to during canal preparation. In the clinical setting, file fracture occurs as a result of a complex combination of flexural and torsional stress that could not be replicated in this study. Instead, each of these stresses was examined separately. Therefore, further studies would be necessary to correlate the data from this study and the clinical performance of the files.

Previous studies suggest that files with higher torsional resistance are more suitable for narrow and/or calcified root canals [37,48,49]. So, within the limitations of this study, TTB may be safer to use when preparing narrow or calcified canals, while RE appears better suited for instrumenting severely curved canals that require more flexibility.

Another point that would have benefited this study is performing differential scanning calorimetry analysis to help describe the crystallographic transformation the heat-treated alloy undergoes at root canal temperature. This could have provided a better explanation of the mechanical features of the tested files [28,31].

The SEM photomicrographs show typical images of fractured topographies due to torsional and cyclic fatigue failure in NiTi files [15,25,31]. The torsional fracture exhibits shear-induced concentric marks at its center, with the dimpling characteristic of brittle fractures, while cyclic fatigue fracture is characterized by linear striations typical of flexural fatigue radiating from an initiation point along with dimpling patterns suggesting ductile fracture.

The fatigue mechanism of NiTi files leads to reduced deflecting load [50], which is considered a deformation-controlled, low-cycle fatigue [51], wherein slow crack propagation has an impact on the file's fatigue life. The fatigue crack growth rate in NiTi alloy has been found to be significantly greater than in other metals of similar strength [52]. This might be due to their machining-induced irregular surface characteristics, which act as stress concentrators [53]. New surface modification technology has shown the potential to enhance the fatigue life of additively manufactured titanium alloys. Laser shock peening utilizes laser-induced plasma shock waves to refine grains and introduce compressive residual stress into the surface layer of the sample. This technology was found to reduce crack growth rates [54]. Another study has shown to improve the fatigue resistance of NiTi wire by increased grain size coupled with stress-control mechanical training [55].

There are certain limitations. In this study, cyclic fatigue was tested under static conditions. However, testing the files under dynamic cyclic fatigue would more closely mimic the clinical practice where the file is axially moved up and down during root canal treatment.

5. Conclusion

This in vitro study was able to show the characterization of blue wire NiTi files that were exposed to cyclic fatigue and torsional fatigue failures. RE is more resistant to cyclic fatigue compared with TTB. While sodium hypochlorite negatively affects the TTB file's resistance to cyclic fatigue, the RE file remains unaffected. On the other hand, the TTB files have superior torsional fatigue resistance compared to the RE files.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/coatings14030361/s1, Table S1: Data set for cyclic fatigue testing; Table S2: Data set for torsional fatigue testing.

Author Contributions: R.M.B., R.A.A. and A.J. designed the study protocol; R.M.B., R.A.A. and A.J. wrote this manuscript; F.N.A., A.A.A., L.S.B. and J.I.A.: prepared material; A.A.A., L.S.B. and J.I.A. and R.M.B. collected data and completed the literature review; A.J. completed the statistical analysis. All authors commented on previous versions of this manuscript. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Princess Nourah Bint Abdulrahman University Researchers Supporting Project number (PNURSP2024R162), Princess Nourah Bint Abdulrahman University, Riyadh, Saudi Arabia.

Institutional Review Board Statement: This in vitro study was exempted according to the Internal Review Board at Princess Nourah Bint Abdulrahman University (IRB no. 22-0442).

Informed Consent Statement: Not applicable.

Data Availability Statement: The datasets generated and analyzed in this study are available as a Supplementary Materials.

Acknowledgments: Princess Nourah Bint Abdulrahman University Researchers Supporting Project number (PNURSP2024R162), Princess Nourah Bint Abdulrahman University, Riyadh, Saudi Arabia.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Estrela, C.; Holland, R.; Estrela, C.R.D.A.; Alencar, A.H.G.; Sousa-Neto, M.D.; Pécora, J.D. Characterization of Successful Root Canal Treatment. *Braz. Dent. J.* **2014**, *25*, 3–11. [CrossRef]
2. 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**, *22*, 195. [CrossRef]
3. Kim, W.; Oh, S.; Ryu, G.J.; Kim, T.H.; Kim, S.J.; Kim, D.H.; Lee, B.N.; Kum, K.Y.; Chang, S.W.; Jang, J.H. Effect of Autoclave Sterilization on Cyclic Fatigue and Torsional Fracture Resistance of NiTi Rotary Instruments. *Odontology* **2020**, *108*, 194–201. [CrossRef]
4. Jamleh, A.; Sadr, A.; Nomura, N.; Yahata, Y.; Ebihara, A.; Hanawa, T.; Tagami, J.; Suda, H. Nano-Indentation Testing of New and Fractured Nickel-Titanium Endodontic Instruments. *Int. Endod. J.* **2012**, *45*, 462–468. [CrossRef]
5. Jamleh, A.; Sadr, A.; Nomura, N.; Ebihara, A.; Yahata, Y.; Hanawa, T.; Tagami, J.; Suda, H. Endodontic Instruments after Torsional Failure: Nanoindentation Test. *Scanning* **2014**, *36*, 437–443. [CrossRef] [PubMed]
6. Zhou, H.-M.; Shen, Y.; Zheng, W.; Li, L.; Zheng, Y.; Haapasalo, M. Mechanical Properties of Controlled Memory and Superelastic Nickel-Titanium Wires Used in the Manufacture of Rotary Endodontic Instruments. *J. Endod.* **2012**, *38*, 1535–1540. [CrossRef] [PubMed]
7. 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. [CrossRef]
8. Yahata, Y.; Yoneyama, T.; Hayashi, Y.; Ebihara, A.; Doi, H.; Hanawa, T.; Suda, H. Effect of Heat Treatment on Transformation Temperatures and Bending Properties of Nickel-Titanium Endodontic Instruments. *Int. Endod. J.* **2009**, *42*, 621–626. [CrossRef] [PubMed]
9. Plotino, G.; Testarelli, L.; Al-Sudani, D.; Pongione, G.; Grande, N.M.; Gambarini, G. Fatigue Resistance of Rotary Instruments Manufactured Using Different Nickel-Titanium Alloys: A Comparative Study. *Odontology* **2014**, *102*, 31–35. [CrossRef] [PubMed]
10. Alapati, S.B.; Brantley, W.A.; Iijima, M.; Schricker, S.R.; Nusstein, J.M.; Li, U.-M.; Svec, T.A. Micro-XRD and Temperature-Modulated DSC Investigation of Nickel-Titanium Rotary Endodontic Instruments. *Dent. Mater.* **2009**, *25*, 1221–1229. [CrossRef]
11. Chan, W.-S.; Gulati, K.; Peters, O.A. Advancing Nitinol: From Heat Treatment to Surface Functionalization for Nickel–Titanium (NiTi) Instruments in Endodontics. *Bioact. Mater.* **2023**, *22*, 91–111. [CrossRef]
12. Zupanc, J.; Vahdat-Pajouh, N.; Schäfer, E. New Thermomechanically Treated NiTi Alloys—A Review. *Int. Endod. J.* **2018**, *51*, 1088–1103. [CrossRef]
13. Gao, Y.; Gutmann, J.L.; Wilkinson, K.; Maxwell, R.; Ammon, D. Evaluation of the Impact of Raw Materials on the Fatigue and Mechanical Properties of ProFile Vortex Rotary Instruments. *J. Endod.* **2012**, *38*, 398–401. [CrossRef]
14. De-Deus, G.; Silva, E.J.N.L.; Vieira, V.T.L.; Belladonna, F.G.; Elias, C.N.; Plotino, G.; Grande, N.M. Blue Thermomechanical Treatment Optimizes Fatigue Resistance and Flexibility of the Reciproc Files. *J. Endod.* **2017**, *43*, 462–466. [CrossRef]
15. Almohareb, R.A.; Barakat, R.; Albakri, A.; Altamimi, M. Effect of Autoclaving Cycles on the Cyclic Fatigue Resistance of Race and Race Evo Nickel-Titanium Endodontic Rotary Files: An In Vitro Study. *Metals* **2021**, *11*, 1947. [CrossRef]
16. Basturk, F.B.; Özyürek, T.; Uslu, G.; Gündoğar, M. Mechanical Properties of the New Generation RACE EVO and R-Motion Nickel–Titanium Instruments. *Materials* **2022**, *15*, 3330. [CrossRef] [PubMed]
17. Tia Tornado Blue File—Tiadent. Available online: http://www.tiadent.com/Tia-Tornado-Blue-File/ (accessed on 24 October 2022).

18. Allahem, Z.; Bendahmash, M.; Almeaither, R.; Alfawaz, H.; Alqedairi, A. Evaluation of the Cyclic Fatigue Resistance of Tia Tornado Blue and Tia Tornado Gold in Curved Canals: In Vitro Study. *Coatings* **2023**, *14*, 15. [CrossRef]
19. Mousavi, S.A.; Norouzi, N.; Memarzadeh, B.; Havaei, S.R.; Yousefshahi, H. Effect of the Temperature of Sodium Hypochlorite on the Cyclic Fatigue Resistance of ProTaper Gold Rotary Files. *Dent. Med. Probl.* **2021**, *58*, 533–537. [CrossRef]
20. Basrani, B.; Haapasalo, M. Update on endodontic irrigating solutions. *Endod. Top.* **2012**, *27*, 74–102. [CrossRef]
21. O'Hoy, P.Y.Z.; Messer, H.H.; Palamara, J.E.A. The Effect of Cleaning Procedures on Fracture Properties and Corrosion of NiTi Files. *Int. Endod. J.* **2003**, *36*, 724–732. [CrossRef] [PubMed]
22. Cheung, G.S.P.; Shen, Y.; Darvell, B.W. Effect of Environment on Low-Cycle Fatigue of a Nickel-Titanium Instrument. *J. Endod.* **2007**, *33*, 1433–1437. [CrossRef]
23. Demiral, M.; Keskin, C.; Inan, U. Effect of Sodium Hypochlorite Activated by Heating on the Cyclic Fatigue Resistance of OneShape Rotary Instruments. *J. Dent. Appl.* **2017**, *4*, 372–375.
24. Gutmann, J.L.; Gao, Y. Alteration in the Inherent Metallic and Surface Properties of Nickel-Titanium Root Canal Instruments to Enhance Performance, Durability and Safety: A Focused Review. *Int. Endod. J.* **2012**, *45*, 113–128. [CrossRef]
25. 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. [CrossRef]
26. Nogueira, D.; Da Silveira Bueno, C.E.; Kato, A.S.; De Martin, A.S.; Pelegrine, R.A.; Da Silva Limoeiro, A.G.; Rocha, D.G.P.; Fontana, C.E. Effect of Immersion in Sodium Hypochlorite on the Cyclic Fatigue Resistance of Three Rotary Instruments. *J. Conserv. Dent.* **2020**, *23*, 554–557. [CrossRef]
27. Vieira, T.M.; Cardoso, R.M.; Alves, N.C.C.; Emanuel Acioly Conrado de Menezes, S.; Batista, S.M.; de Andrade Silva, S.; Velozo, C.; de Albuquerque, D.S.; de Melo Monteiro, G.Q. Cyclic Fatigue Resistance of Blue Heat-Treated Instruments at Different Temperatures. *Int. J. Biomater.* **2021**, *2021*, 5584766. [CrossRef]
28. Peters, O.A.; Chien, P.Y.-H.; Armitt, K.; Macorra, J.C.; Arias, A. Testing Cyclic Fatigue Resistance of Nickel Titanium Rotary Endodontic Instruments: A Validation Study for a Minimum Quality Criterion in a Standardized Environment. *Front. Dent. Med.* **2021**, *2*, 744809. [CrossRef]
29. Alqedairi, A.; Alfawaz, H.; bin Rabba, A.; Almutairi, A.; Alnafaiy, S.; Khan Mohammed, M. Failure Analysis and Reliability of Ni–Ti-Based Dental Rotary Files Subjected to Cyclic Fatigue. *Metals* **2018**, *8*, 36. [CrossRef]
30. Faus-Llácer, V.; Hamoud-Kharrat, N.; Marhuenda Ramos, M.T.; Faus-Matoses, I.; Zubizarreta-Macho, Á.; Ruiz Sánchez, C.; Faus-Matoses, V. Influence of the Geometrical Cross-Section Design on the Dynamic Cyclic Fatigue Resistance of NiTi Endodontic Rotary Files—An In Vitro Study. *J. Clin. Med.* **2021**, *10*, 4713. [CrossRef]
31. Zanza, A.; Russo, P.; Di Matteo, P.; Reda, R.; Di Nardo, D.; Gambarini, G.; Testarelli, L. Mechanical Properties and Metallurgical Features of Two Similar Endodontic Rotary Instruments with Different Heat Treatments (FireWire^{TM} and Gold). *Sci. Prog.* **2022**, *105*, 00368504221103763. [CrossRef] [PubMed]
32. Almohareb, R.A.; Barakat, R.; Albohairy, F. New Heat-Treated vs Electropolished Nickel-Titanium Instruments Used in Root Canal Treatment: Influence of Autoclave Sterilization on Surface Roughness. *PLoS ONE* **2022**, *17*, e0265226. [CrossRef]
33. Alfawaz, H.; Alqedairi, A.; Alsharekh, H.; Almuzaini, E.; Alzahrani, S.; Jamleh, A. Effects of Sodium Hypochlorite Concentration and Temperature on the Cyclic Fatigue Resistance of Heat-Treated Nickel-Titanium Rotary Instruments. *J. Endod.* **2018**, *44*, 1563–1566. [CrossRef]
34. Topçuoğlu, H.S.; Pala, K.; Aktı, A.; Düzgün, S.; Topçuoğlu, G. Cyclic Fatigue Resistance of D-RaCe, ProTaper, and Mtwo Nickel–Titanium Retreatment Instruments after Immersion in Sodium Hypochlorite. *Clin. Oral Investig.* **2016**, *20*, 1175–1179. [CrossRef]
35. Uslu, G.; Özyürek, T.; Yılmaz, K.; Plotino, G. Effect of Dynamic Immersion in Sodium Hypochlorite and EDTA Solutions on Cyclic Fatigue Resistance of WaveOne and WaveOne Gold Reciprocating Nickel-Titanium Files. *J. Endod.* **2018**, *44*, 834–837. [CrossRef] [PubMed]
36. Pedullà, E.; Grande, N.M.; Plotino, G.; Palermo, F.; Gambarini, G.; Rapisarda, E. Cyclic Fatigue Resistance of Two Reciprocating Nickel-Titanium Instruments after Immersion in Sodium Hypochlorite. *Int. Endod. J.* **2013**, *46*, 155–159. [CrossRef]
37. Kaval, M.E.; Capar, I.D.; Ertas, H. Evaluation of the Cyclic Fatigue and Torsional Resistance of Novel Nickel-Titanium Rotary Files with Various Alloy Properties. *J. Endod.* **2016**, *42*, 1840–1843. [CrossRef] [PubMed]
38. Silva, E.J.N.L.; Giraldes, J.F.N.; de Lima, C.O.; Vieira, V.T.L.; Elias, C.N.; Antunes, H.S. Influence of Heat Treatment on Torsional Resistance and Surface Roughness of Nickel-Titanium Instruments. *Int. Endod. J.* **2019**, *52*, 1645–1651. [CrossRef] [PubMed]
39. Baek, S.H.; Lee, C.J.; Versluis, A.; Kim, B.M.; Lee, W.; Kim, H.C. Comparison of Torsional Stiffness of Nickel-Titanium Rotary Files with Different Geometric Characteristics. *J. Endod.* **2011**, *37*, 1283–1286. [CrossRef]
40. FKG Race_Evo | FKG Dentaire. Available online: https://www.fkg.ch/sites/default/files/FKG_RACEEVO4%25_Protocolcard_EN_WEB_202110.pdf (accessed on 18 June 2021).
41. Otubo, J.; Rigo, O.D.; Coelho, A.A.; Neto, C.M.; Mei, P.R. The Influence of Carbon and Oxygen Content on the Martensitic Transformation Temperatures and Enthalpies of NiTi Shape Memory Alloy. *Mater. Sci. Eng. A* **2008**, *481–482*, 639–642. [CrossRef]
42. Faus-Matoses, V.; Pérez García, R.; Faus-Llácer, V.; Faus-Matoses, I.; Alonso Ezpeleta, Ó.; Albaladejo Martínez, A.; Zubizarreta-Macho, Á. Comparative Study of the SEM Evaluation, EDX Assessment, Morphometric Analysis, and Cyclic Fatigue Resistance of Three Novel Brands of NiTi Alloy Endodontic Files. *Int. J. Environ. Res. Public Health* **2022**, *19*, 4414. [CrossRef]

43. Jia, C.; Akbarpour, M.R.; Ahmadi Gharamaleki, M.; Ebadzadeh, T.; Kim, H.S. Synthesis and Characterization of Novel NiTi–Ni3Ti/SiC Nanocomposites Prepared by Mechanical Alloying and Microwave-Assisted Sintering Process. *Ceram. Int.* **2023**, *49*, 23358–23366. [CrossRef]
44. Salvetr, P.; Školáková, A.; Novák, P.; Vavřík, J. Effect of Si Addition on Martensitic Transformation and Microstructure of NiTiSi Shape Memory Alloys. *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.* **2020**, *51*, 4434–4438. [CrossRef]
45. Chi, C.-W.; Deng, Y.-L.; Lee, J.-W.; Lin, C.-P. Fracture Resistance of Dental Nickel–Titanium Rotary Instruments with Novel Surface Treatment: Thin Film Metallic Glass Coating. *J. Formos. Med. Assoc.* **2017**, *116*, 373–379. [CrossRef] [PubMed]
46. Shahmir, H.; Kiani, F.; Baradari, S.; Resnina, N.; Bikbaev, R. Role of Zr and Thermomechanical Treatment on Phase Transformation and Functional Properties of NiTi-Based Shape Memory Alloys. *J. Mater. Res. Technol.* **2023**, *26*, 3110–3117. [CrossRef]
47. Lepule, M.L.; Obadele, B.A.; Andrews, A.; Olubambi, P.A. Corrosion and Wear Behaviour of ZrO_2 Modified NiTi Coatings on AISI 316 Stainless Steel. *Surf. Coat. Technol.* **2015**, *261*, 21–27. [CrossRef]
48. Oh, S.; Kim, T.-H.; Chang, S.W. Mechanical Properties of NiTi Rotary Files Fabricated through Gold-Wire, CM-Wire, T-Wire, and R-Phase Heat Treatment. *Appl. Sci.* **2023**, *13*, 3604. [CrossRef]
49. Pereira, É.S.J.; Viana, A.C.D.; Buono, V.T.L.; Peters, O.A.; de Azevedo Bahia, M.G. Behavior of Nickel-Titanium Instruments Manufactured with Different Thermal Treatments. *J. Endod.* **2015**, *41*, 67–71. [CrossRef] [PubMed]
50. Jamleh, A.; Kobayashi, C.; Yahata, Y.; Ebihara, A.; Suda, H. Deflecting Load of Nickel Titanium Rotary Instruments during Cyclic Fatigue. *Dent. Mater. J.* **2012**, *31*, 389–393. [CrossRef]
51. Duerig, T.; Pelton, A.; Stöckel, D. An Overview of Nitinol Medical Applications. *Mater. Sci. Eng. A* **1999**, *273–275*, 149–160. [CrossRef]
52. Dauskardt, R.; Duerig, T.; Ritchie, R. Effects of In Situ Phase Transformation on Fatigue-Crack Propagation in Titanium—Nickel Shape-Memory Alloys. *Proc. Int. Meet. Adv. Mater.* **1989**, *9*, 243–249.
53. Bahia, M.G.A.; Melo, M.C.C.; Buono, V.T.L. Influence of Simulated Clinical Use on the Torsional Behavior of Nickel-Titanium Rotary Endodontic Instruments. *Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod.* **2006**, *101*, 675–680. [CrossRef] [PubMed]
54. Zhang, Y.; Guo, W.; Shi, J.; Chi, J.; Chen, G.; Han, G.; Zhang, H. Improved Rotating Bending Fatigue Performance of Laser Directed Energy Deposited Ti6Al4V Alloys by Laser Shock Peening. *J. Alloys Compd.* **2024**, *980*, 173664. [CrossRef]
55. Chen, P.; Cai, X.; Liu, Y.; Wang, Z.; Jin, M.; Jin, X. Combined Effects of Grain Size and Training on Fatigue Resistance of Nanocrystalline NiTi Shape Memory Alloy Wires. *Int. J. Fatigue* **2023**, *168*, 107461. [CrossRef]