Journal of Dental Sciences 19 (2024) 929–936

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.e-jds.com

Original Article

Phase transformation behavior and mechanical properties of HyFlex EDM nickel-titanium endodontic rotary instrument: Evaluation at body temperature

Shunsuke Kimura, Arata Ebihara*, Keiichiro Maki, Moe Sandar Kyaw, Yuka Kasuga, Satoshi Omori, Takashi Okiji

Department of Pulp Biology and Endodontics, Division of Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University (TMDU), Tokyo, Japan

Received 29 June 2023; Final revision received 31 July 2023
Available online 11 August 2023

KEYWORDS
Differential scanning calorimetry;
Fatigue fracture;
Flexibility;
Nickel titanium alloy;
Root canal preparation

Abstract *Background/purpose:* Temperature-dependent phase compositional changes influence the mechanical properties of heat-treated nickel-titanium (NiTi) rotary instruments. This study evaluated the phase composition, bending properties, and cyclic fatigue resistance of HyFlex EDM NiTi rotary instruments against differently heat-treated and non-heat-treated NiTi instruments at body temperature (BT).
Materials and methods: HyFlex EDM OneFile (EDM) instruments, heat-treated HyFlex CM (CM) and Twisted File (TF) instruments, and non-heat-treated K3 instruments (size #25/.08) were subjected to differential scanning calorimetry, and the martensitic, R-phase, and reverse transformation starting and finishing temperatures were determined. A cantilever bending test and a cyclic fatigue test were conducted at BT (37 °C $\pm$ 1.0 °C), and the bending load and number of cycles to failure (NCF) were recorded. Statistical analysis was performed using Kruskal–Wallis and Mann–Whitney U tests ($\alpha = 0.05$).
Results: TF and K3 had reverse transformation finishing temperatures lower than BT, while those for EDM and CM were higher than BT. The bending loads at a 0.5 mm deflection were in the order of EDM $<$ TF $<$ CM $<$ K3 ($P < 0.05$), and those at a 2.0 mm deflection were EDM $<$ CM and TF $<$ K3 ($P < 0.05$). EDM had the highest NCF among the four instruments ($P < 0.05$).
Conclusion: The EDM instrument had a reverse transformation finishing temperature higher than BT indicating its martensite/R-phase composition at BT. The EDM instrument had superior flexibility and greater resistance to cyclic fatigue than the CM, TF, and K3 instruments at BT.

* Corresponding author. Department of Pulp Biology and Endodontics, Division of Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University (TMDU), 1-5-45, Yushima, Bunkyo-ku, Tokyo 113-8549, Japan.
E-mail address: a.ebihara.endo@tmd.ac.jp (A. Ebihara).

https://doi.org/10.1016/j.jds.2023.07.039

Introduction

Nickel–titanium (NiTi) rotary instruments have several advantages for shaping curved canals, such as greater flexibility[1] and better ability to maintain the original canal shape[2] than stainless steel files. However, unexpected intracanal separation because of torsion or flexural fatigue is a major concern for their clinical use.[3] To prevent such issues, attempts have been made to improve the fracture resistance of NiTi instruments using various strategies, including modifications in geometry,[4] kinematics,[5] and metallurgy.[1]

Heat treatment changes the phase transformation temperature of NiTi alloys by inducing crystal structural changes via the transformation from the austenitic phase to the softer and more ductile martensitic phase and R-phase.[6] Thus, the phase transformation behavior of a NiTi rotary instrument is crucial in determining the instrument's flexibility and fracture resistance. The phase composition status of NiTi instruments is temperature dependent,[7] indicating that their mechanical performance should be tested at an intraoral temperature. However, most studies have been conducted at room temperature,[8,9] which may limit the clinical relevance of their results.

HyFlex EDM instruments (Coltene/Whaledent, AG, Altstatten, Switzerland), a modification of HyFlex CM instruments (Coltene/Whaledent),[10] are manufactured using electro-discharge machining (EDM) of the heat-treated controlled memory (CM) wire. EDM is a non-contact thermal erosion process that partially melts and evaporates the surface of the wire using high-frequency spark discharges.[11] HyFlex EDM has a greater cyclic fatigue resistance than some rotary and reciprocating NiTi instruments,[12] exhibiting four times more number of cycles to failure (NCF) than HyFlex CM.[13] The Twisted File (TF, SybronEndo, Orange, CA, USA) is another heat-treated NiTi instrument that is manufactured by twisting R-phase NiTi wire, and it shows better fatigue resistance than non-heat-treated NiTi instruments.[14–17] K3 (SybronEndo) is a traditional non-heat-treated NiTi instrument manufactured with a grinding process and shows lower cyclic fatigue resistance than TF.[18]

The flexibility of NiTi rotary instruments is a key factor that contributes to their cyclic fatigue resistance.[19] Factors that may determine the flexibility include the amount of martensite and/or R-phase in the alloy, which is related to the phase transformation behavior of NiTi alloys and thus is temperature dependent. However, few studies have investigated both the cyclic fatigue resistance and bending properties of heat-treated NiTi instruments at body temperature in relation to their phase transformation behavior.[19] In particular, HyFlex EDM instrument needs further investigation to assure its high cyclic fatigue resistance[12,13] under a clinically-relevant temperature setting. Thus, the aim of this study was to evaluate the HyFlex EDM instrument against other heat-treated and non-heat-treated NiTi instruments in terms of thermal behavior, bending properties, and cyclic fatigue resistance at body temperature. The null hypothesis was that there are no differences in the bending properties and cyclic fatigue resistance among the NiTi instruments investigated.

Materials and methods

HyFlex EDM OneFile, HyFlex CM, TF, and K3 instruments with the same tip size (#25) and 0.08 taper were used.

Sample size estimation

In accordance with a previous study,[19] G*Power (Version 3.1.9.7; Heine Heinrich Universität, Düsseldorf, Germany) was employed with an effect size of 0.6, α error of 0.05, and power of 0.95, resulting in a sample size of 10 per group.

Differential scanning calorimetry

In accordance with a previous study,[19] differential scanning calorimetry (DSC) was used to evaluate the phase transformation behavior of the four instruments (n = 5, each). Briefly, the blade portion of each instrument was cut into 2- to 3-mm long sections. Specimens weighing 20 mg each were measured using a differential scanning calorimeter (DSC-60; Shimadzu, Kyoto, Japan). Liquid nitrogen acted as the coolant, and the heating and cooling rates were set at 0.33 °C/s. The temperature was increased to 100 °C from room temperature, and then decreased to −100 °C to obtain the cooling curve. The temperature was then increased to 100 °C to obtain the heating curve.

Phase transformation temperatures were obtained from the intersections between extrapolations of the baseline and the maximum gradient line of the lambda-type DSC curve.[14,19–21] The martensitic transformation starting and finishing temperatures (Ms and Mf, respectively), the R-phase transformation starting and finishing temperatures (Rs and Rf, respectively), and the reverse transformation starting and finishing temperatures (As and Af, respectively) were determined.

Bending test

EDM, CM, TF, and K3 instruments (n = 10, each) were tested using a cantilever bending test based on previous studies.[14,20,22] A universal testing machine (Autograph AG-IS; Shimadzu) was used to apply and record the load. After cutting off the handle, each specimen was clamped 7.0 mm from the tip and the loading point was set 2.0 mm from the tip (Fig. 1). The loading speed was 1.0 mm/min, and once the deflection reached 3.0 mm, the unloading process was started. The bending load values were continuously recorded, and those of 0.5 mm and 2.0 mm were further analyzed as representative points of the elastic and superelastic ranges, respectively. The temperatures of the experimental specimens and device were

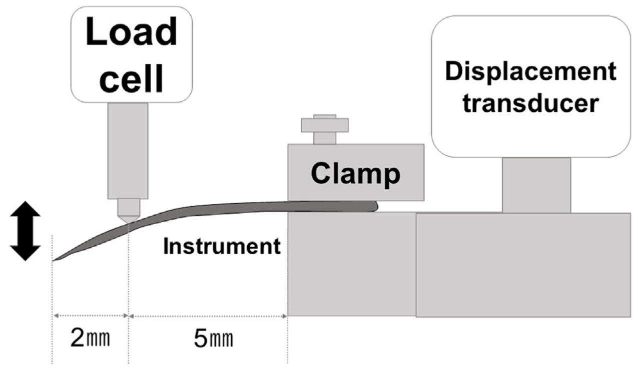

Figure 1 Schematic drawing of the cantilever bending test device.

maintained at 37 °C ± 1.0 °C (referred to as body temperature; BT) by maintaining the experimental setup in a transparent glass container equipped with a temperature-adjustable fan heater (RKC Instrument Inc., Tokyo, Japan).

Cyclic fatigue test

Ten specimens of each instrument were tested. The experimental setup was based on previous studies using a stainless steel three-pin device.[19,21,22] The three pins were placed to curve the instrument (Fig. 2). The No. 3 pin was placed 2 mm from the tip of the instrument, and the No. 2 and No. 1 pins were placed 5 mm toward the base of the instrument. The instrument curvature was accurately reproduced by horizontally adjusting the No. 3 pin to the left. Silicone oil (KF-96-100CS; ShinEtsu Chemical, Tokyo, Japan) was used as a lubricant to minimize friction and heat generation. The temperature of the instrument and apparatus was maintained at BT by housing the experimental setup in a transparent glass container equipped with a temperature-adjustable fan heater (Omron-E5C4; OMRON Corporation, Kyoto, Japan).

The instrument was fixed using the No. 1 and No. 2 pins, and the instrument was protruded 2 mm beyond the No. 3 pin. The instrument curvature was 38° with a curvature radius of 5 mm. The instrument was rotated at 300 rpm using an endodontic motor (Dentaport ZX; J Morita, Kyoto, Japan).

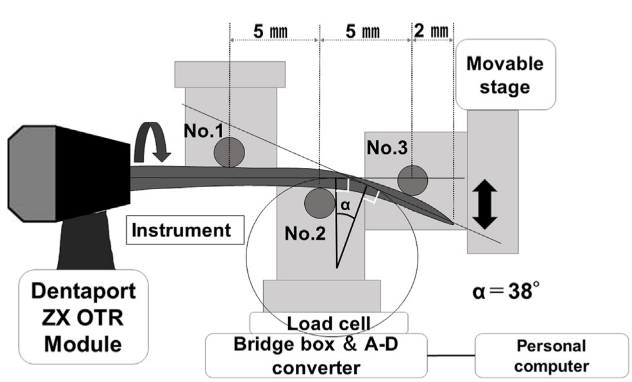

Figure 2 Schematic drawing of the cyclic fatigue test device. No. 1, No. 2 and No. 3: three pins used to curve the instrument.

A load cell (LUR-A-50NSA1; Kyowa Electronic Instruments, Tokyo, Japan) was fixed to the No. 2 pin to determine the magnitude of the deflection load imposed by the instrument during rotation. The output of the load cell was connected to a personal computer (VAIO, VGN-FE92HS; Sony, Tokyo, Japan) via an analog-to-digital converter with a bridge box (TUSB-S01LC; Turtle Industry, Tsuchiura, Japan). The time to fracture was measured and NCF values were determined as the number of revolutions (rpm) × time to fracture (minutes).

The fractured surface of the separated NiTi instruments was investigated with a scanning electron microscope (S-3400; Hitachi, Tokyo, Japan) at a 15 kV accelerating voltage to determine the fracture mode.

Data analysis

Statistical analysis was conducted using statistical software (SPSS v27; IBM Corp, Armonk, NY, USA). A Kruskal—Wallis test with a post hoc Mann—Whitney *U* test and Bonferroni correction was used to detect the difference of the bending load and NCF values among the tested instruments. The statistical significance level was set at 5%.

Results

Phase transformation temperatures

Fig. 3 shows typical DSC curves obtained from each group. The upper peak indicates the exothermic reaction accompanying the R-phase/martensitic transformation from the austenitic phase during the cooling process. The lower peak indicates the endothermic reaction caused by the reverse transformation during the heating process. The EDM instrument had two clear exothermic peaks and thus, R_s and R_f were obtained. The K3 and TF instruments had a single exothermic peak and the CM instrument had multiple exothermic peaks that were not clearly separate; thus, R_s and R_f were not determined. The M_s and M_f of the CM instrument were determined from the tangent line where the DSC curve deviates from and returns to the baseline. During the endothermic process, the EDM and K3 instruments had a single peak, whereas the CM and TF instruments had two overlapping peaks.

The phase transformation temperatures of all groups are shown in Table 1. EDM and CM instruments had an A_f greater than BT, while the TF and K3 instruments had an A_f less than BT.

Bending load values

Fig. 4 shows typical stress-induced deflection curves for each instrument. EDM and CM instruments had typical deflection curves of shape memory NiTi alloys with permanent deformation. TF and K3 instruments had typical deflection curves of a superelastic NiTi alloy.

As shown in Table 2, the bending load values at 0.5 mm deflection, representing the elastic range, were in the order of EDM < TF < CM < K3 ($P < 0.05$), and those at

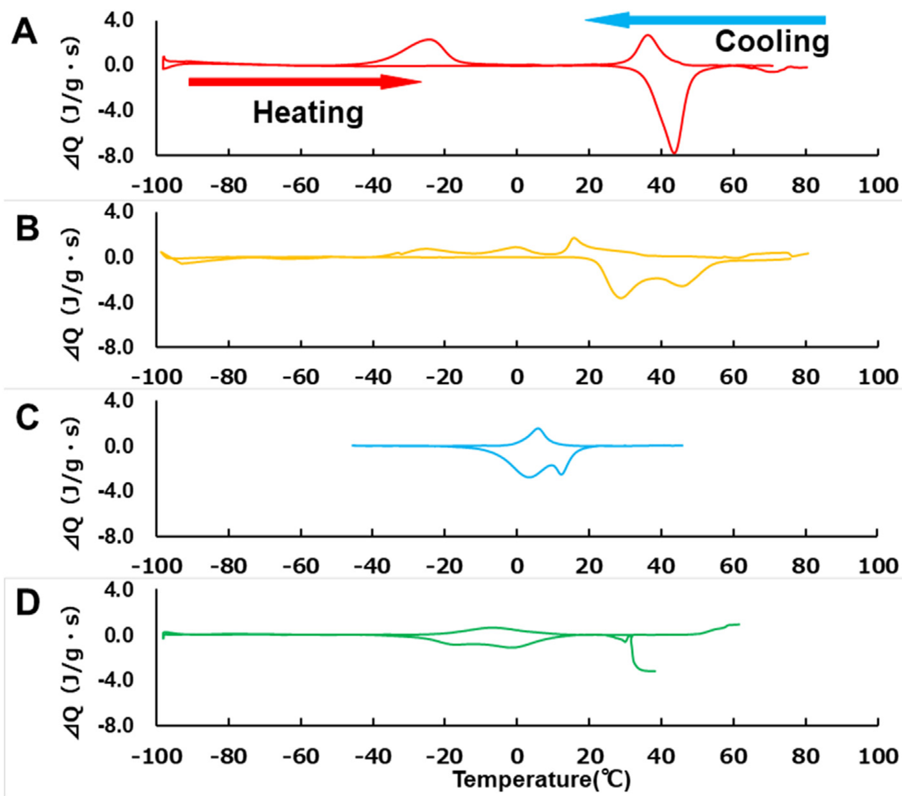

Figure 3 Typical DSC curves for (A) HyFlex EDM, (B) HyFlex CM, (C) Twisted File, and (D) K3 instruments.

Table 1 Phase transformation temperatures of the instruments investigated.

	Ms (°C)	*Mf* (°C)	*Rs* (°C)	*Rf* (°C)	*As* (°C)	*Af* (°C)
EDM	−15.79 ± 1.66	−40.4 ± 0.53	42.6 ± 0.46	30.84 ± 0.21	36.7 ± 0.57	49.9 ± 1.0
CM	36.5 ± 0.73	−32.4 ± 1.41	—	—	29.1 ± 1.59	57.2 ± 2.11
TF	9.1 ± 1.22	−2.4 ± 2.11	—	—	−8.4 ± 1.61	15.5 ± 0.66
K3	7.1 ± 2.65	−22.3 ± 3.4	—	—	−27.1 ± 0.57	13.1 ± 2.46

Data are mean and SD (n = 5).
Abbreviations: *Af*: reverse transformation starting and finishing temperature, *As*: reverse transformation starting temperature, *Mf*: martensitic transformation finishing temperature.
Ms, martensitic transformation starting temperature, *Rf*: R-phase transformation finishing temperature, *Rs*: R-phase transformation starting temperature, EDM: HyFlex EDM, CM: HyFlex CM,TF: Twisted File.

2.0 mm deflection, representing the superelastic range, were EDM < CM and TF < K3 ($P < 0.05$).

Cyclic fatigue test

As shown in Table 2, the EDM instrument had the highest NCF among the tested instruments ($P < 0.05$), and the NCF for the TF instrument was significantly higher than for the K3 instrument. The NCF for the CM instrument was not significantly different than that from the TF and K3 instruments ($P > 0.05$).

Representative SEM images (Fig. 5) show typical features of cyclic fatigue failure, including crack-initiating areas and dimples.

Discussion

In this study, the phase transformation temperature, bending loads, and NCF values of EDM and other NiTi rotary instruments were compared at a clinically-relevant BT condition. Overall, the EDM instrument showed a significantly lower bending load and higher NCF than the other investigated instruments. Thus, the null hypothesis was rejected.

The NiTi alloy phase composition changes depending on the environmental temperature, which affects the mechanical properties of the alloy at specific temperatures.[23–25] The phase transformation behavior of NiTi alloys is sensitive to several factors, including the atomic composition, heat treatment, and manufacturing

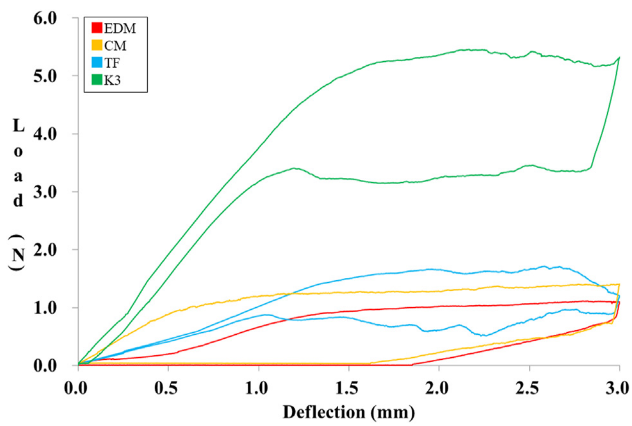

Figure 4 Typical stress-induced deflection curves for the instruments investigated. CM: HyFlex CM, EDM: HyFlex EDM, TF: Twisted File.

Table 2 Bending load values, number of cycles to failure (NCF), and fracture length for EDM, CM, TF, and K3 instruments (n = 10).

Group	Bending load value		NCF
	0.5-mm deflection (N)	2.0-mm deflection (N)	
EDM	0.17 (0.17—0.19)^{a}	0.99 (0.63—1.05)^{a}	4203 (2774—4813)^{a}
CM	0.93 (0.83—0.99)^{b}	1.45 (1.40—1.68)^{b}	545 (323—653) ^{bc}
TF	0.48 (0.39—0.52)^{c}	1.46 (1.25—1.65)^{b}	517 (490—543)^{b}
K3	2.62 (2.57—2.79)^{d}	7.14 (6.86—7.35)^{c}	354 (312—378)^{c}

Data show medians and interquartile ranges. Different superscript letters in the same column indicate statistical differences among groups ($p < 0.05$).
Abbreviations: CM: HyFlex CM, EDM:HyFlex EDM, TF: Twisted File, NCF: number of cycles to failure.

process.[26,27] In general, martensitic instruments are soft, ductile, and have greater cyclic fatigue resistance than austenitic NiTi instruments.[28] Understanding the phase transformation behavior of a NiTi rotary instrument, particularly under BT, is important because the phase composition can be critical in determining the instrument's efficiency and safety under clinical conditions.

The DSC results showed various phase transition patterns, indicating the tested instruments had different phase states at BT (Fig. 3, Table 1). The R*s* of the EDM instrument (36.7 °C ± 0.57 °C) was nearly identical to BT, indicating the instrument was rich in the R-phase, an intermediate phase with better resistance to cyclic fatigue than austenite.[18] This agrees with earlier studies.[11,29,30] The CM instrument may be a mixed phase of austenite and martensite at BT[11] because the A*s* (29.1 °C ± 1.59 °C) and A*f* (57.2 °C ± 2.11 °C) were lower and higher, respectively, than BT. The CM instrument is reported to have two exothermic peaks, suggesting the existence of the R-phase[31]; however, this was not demonstrated in this study likely because of different experimental conditions. The higher A*s* temperature of the EDM instrument than that of the CM instrument indicate that the EDM process induces a temperature shift to produce more martensite and R-phase than the conventional grinding process, presumably by causing precipitation of Ni_4Ti_3.[8,11]

The TF and K3 instruments may be mainly austenitic at BT because the M*s* and A*f* were less than BT, which is consistent with earlier studies.[14,15] The presence of two endothermic peaks in the TF instrument suggests that the reverse transformation of the alloy to austenite might proceed via the formation of the R-phase.

Most published studies reported bending and cyclic fatigue testing of NiTi rotary instruments at room temperature,[32,33] which may limit the clinical relevance of the results because of the difference in the phase composition and resulting mechanical properties of the instruments at room temperature and BT.[6,34,35] In this regard, the DSC analysis in this study suggests that the CM instrument is primarily martensitic at room temperature; however, it is a mixed phase of martensite and austenite at BT. This implies that the CM instrument may be more flexible and fatigue resistant at room temperature, leading to overestimation of its clinical performance if such properties are analyzed at room temperature.

The elastic moduli of NiTi alloys differ depending on the constituent phase; the martensitic phase has lower moduli than the austenite phase, and the moduli changes remarkably near the transformation temperature.[21] According to the presented bending test results, the EDM instrument showed the lowest load value among all instruments tested. Such high flexibility of the EDM instrument, likely because of its dominant R-phase composition at BT, is consistent with previous studies.[36,37] The EDM process might also affect the bending load values.[13,30]

The bending load value of the TF instrument was significantly smaller than that of the CM instrument in the elastic range, while the two instruments exhibited similar values in the superelastic range. The TF instrument may contain a certain amount of the R-phase, which has a lower elastic modulus than martensite and austenite. Moreover, the M*s* of the TF instrument was less than BT, which impedes the phase transformation, and more stress is required for stress-induced martensitic transformation in the superelastic range.[14,20]

Another factor that influences the bending load is the geometry of the instruments, including the cross-sectional configuration and area, pitch length, and taper.[21,22] The smaller triangular cross-sectional area of the TF instrument may contribute to the smaller load that is sustained in the elastic range. The K3 instrument sustained the highest load among the instruments tested. This agrees with earlier findings[14,15] and can be explained by the cross-sectional area of the K3 instrument, which is twice that of the TF instrument.[15] Additionally, the K3 instruments had the lowest transformation temperature and austenitic phase composition.

In this study, a cyclic fatigue test was conducted by applying constant load to the NiTi instruments under a flexed state, similar to previous studies.[14,19,21] The scanning electron microscopic analysis showed typical

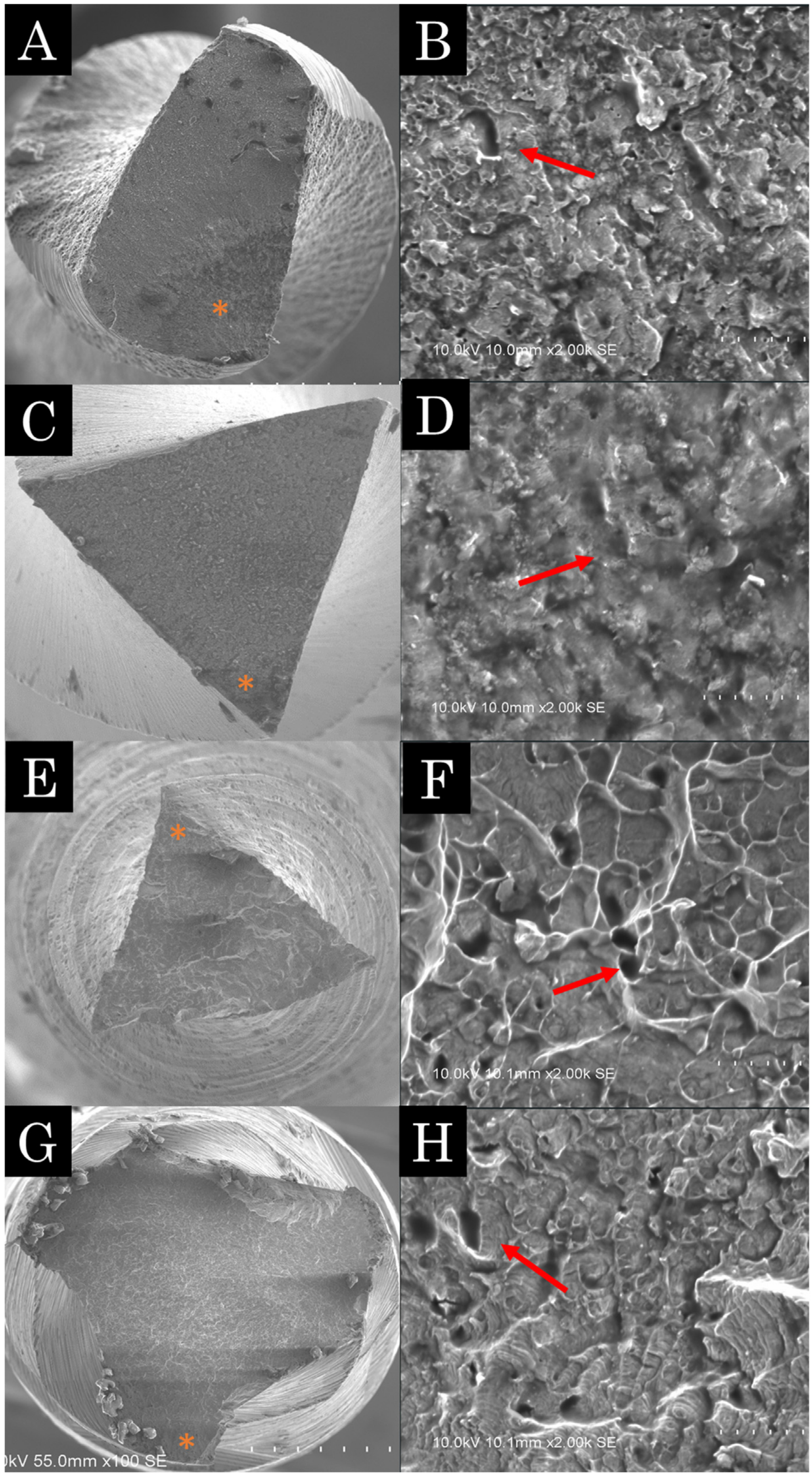

Figure 5 Representative scanning electron microscopic images of the fractured surface of a broken fragment of (A, B) HyFlex EDM, (C, D) HyFlex CM, (E, F) Twisted File, and (G, H) K3 instruments after the cyclic fatigue test. Asterisks indicate the crack initiating area. Red arrows indicate dimples.

fractographic appearances of cyclic fatigue fracture characterized by crack initiation areas at the cutting edge and dimples on the fracture surface indicating ductile repture.[12,13,17,38] According to the load—deflection curve, the range of deflection under the cyclic fatigue test conditions may have been in the superelastic range. The EDM instrument had the highest NCF, which is in line with several studies showing the superior cyclic fatigue resistance of EDM.[12,13,29,30,36,37,39,40] The phase composition of the EDM instrument at BT (martensitic or R-phase) may be important because instruments with greater flexibility tend to exhibit higher cyclic fatigue resistance.[6,9,30,37] Moreover, the EDM procedure created a few machining grooves that are common in conventional manufacturing methods, and such special surface conditions may increase the cyclic fatigue resistance of the EDM instrument.[39] Additionally, the EDM instrument has a crater-like surface texture, which may contribute to blocking crack propagation.[40]

This study had several limitations. First, there are inherent differences between laboratory and clinical conditions, and the tests employed may be regarded as screening tests that cannot be directly extrapolated to the clinical performance of the instruments tested.[41] Second, similar to most studies employing commercially available NiTi instruments, the effect of various geometric factors were not eliminated, and thus it was difficult to attribute differences among groups to only the metallurgical factors.[41]

In conclusion, the EDM instrument showed better flexibility and cyclic fatigue resistance than differently heat-treated and non-heat-treated NiTi rotary instruments at BT. The EDM instrument had a reverse transformation finishing temperature higher than BT, indicating the R-phase-rich composition of the EDM instrument at BT. Within the limitations of these in vitro findings, the EDM instrument appears suitable for use in curved canals.

Declaration of competing interest

The authors declare no conflicts of interest.

Acknowledgements

This study was supported by grants-in-aid for Scientific Research (Nos. 21K09890ZA, 21K16963ZA, 22K17051ZA) from the Japan Society for the Promotion of Sciences. We thank Ashleigh Cooper, PhD, from Edanz (https://jp.edanz.com/ac) for editing a draft of this manuscript.

References

1. Ebihara A, Yahata Y, Miyara K, Nakano K, Hayashi Y, Suda H. Heat treatment of nickel-titanium rotary endodontic instruments: effects on bending properties and shaping abilities. *Int Endod J* 2011;44:843—9.
2. Sonntag D, Guntermann A, Kim SK, Stachniss V. Root canal shaping with manual stainless steel files and rotary Ni-Ti files performed by students. *Int Endod J* 2003;36:246—55.
3. Parashos P, Messer HH. Rotary NiTi instrument fracture and its consequences. *J Endod* 2006;32:1031—43.
4. Ha JH, Cheung GS, Versluis A, Lee CJ, Kwak SW, Kim HC. 'Screw-in' tendency of rotary nickel-titanium files due to design geometry. *Int Endod J* 2015;48:666—72.
5. Kimura S, Ebihara A, Maki K, Nishijo M, Tokita D, Okiji T. Effect of optimum torque reverse motion on torque and force generation during root canal instrumentation with crown-down and single-length techniques. *J Endod* 2020;46:232—7.
6. Zupanc J, Vahdat-Pajouh N, Schäfer E. New thermomechanically treated NiTi alloys — a review. *Int Endod J* 2018;51:1088—103.
7. Shen Y, Zhou HM, Wang Z, Campbell L, Zheng YF, Haapasalo M. Phase transformation behavior and mechanical properties of thermomechanically treated K3XF nickel-titanium instruments. *J Endod* 2013;39:919—23.
8. Hülsmann M, Donnermeyer D, Schäfer E. A critical appraisal of studies on cyclic fatigue resistance of engine-driven endodontic instruments. *Int Endod J* 2019;52:1427—45.
9. Schäfer E, Bürklein S, Donnermeyer D. A critical analysis of research methods and experimental models to study the physical properties of NiTi instruments and their fracture characteristics. *Int Endod J* 2022;55:72—94.
10. Santos LdA, Bahia MGdA, Las Casas EBd, Buono VTL. Comparison of the mechanical behavior between controlled memory and superelastic nickel-titanium files via finite element analysis. *J Endod* 2013;39:1444—7.
11. Iacono F, Pirani C, Generali L, et al. Structural analysis of HyFlex EDM instruments. *Int Endod J* 2017;50:303—13.
12. Pedullà E, Lo Savio F, Boninelli S, et al. Torsional and cyclic fatigue resistance of a new nickel-titanium instrument manufactured by electrical discharge machining. *J Endod* 2016;42:156—9.
13. Pirani C, Iacono F, Generali L, et al. HyFlex EDM: superficial features, metallurgical analysis and fatigue resistance of innovative electro discharge machined NiTi rotary instruments. *Int Endod J* 2016;49:483—93.
14. Hou X, Yahata Y, Hayashi Y, Ebihara A, Hanawa T, Suda H. Phase transformation behaviour and bending property of twisted nickel—titanium endodontic instruments. *Int Endod J* 2011;44:253—8.
15. Gambarini G, Grande NM, Plotino G, et al. Fatigue Resistance of engine-driven rotary nickel-titanium instruments produced by new manufacturing methods. *J Endod* 2008;34:1003—5.
16. Oh SR, Chang SW, Lee Y, et al. A comparison of nickel-titanium rotary instruments manufactured using different methods and cross-sectional areas: ability to resist cyclic fatigue. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2010;109:622—8.
17. Kim HC, Yum J, Hur B, Cheung GSP. Cyclic fatigue and fracture characteristics of ground and twisted nickel-titanium rotary files. *J Endod* 2010;36:147—52.
18. Pérez-Higueras JJ, Arias A, de la Macorra JC. Cyclic fatigue resistance of K3, K3XF, and Twisted File nickel-titanium files under continuous rotation or reciprocating motion. *J Endod* 2013;39:1585—8.
19. Miyara K, Yahata Y, Hayashi Y, et al. The influence of heat treatment on the mechanical properties of Ni-Ti file materials. *Dent Mater J* 2014;33:27—31.
20. Miyai K, Ebihara A, Hayashi Y, Doi H, Suda H, Yoneyama T. Influence of phase transformation on the torsional and bending properties of nickel-titanium rotary endodontic instruments. *Int Endod J* 2006;39:119—26.
21. Yahata Y, Yoneyama T, Hayashi Y, et al. Effect of heat treatment on transformation temperatures and bending properties of nickel—titanium endodontic instruments. *Int Endod J* 2009;42:621—6.
22. 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* 2014;33:539—44.

23. Yoneyama T, Doi H, Hamanaka H, Yamamoto M, Kuroda T. Bending properties and transformation temperatures of heat treated Ni-Ti alloy wire for orthodontic appliances. *J Biomed Mater Res* 1993;27:399—402.
24. Huang X, Liu Y. Effect of annealing on the transformation behavior and superelasticity of NiTi shape memory alloy. *Scripta Mater* 2001;45:153—60.
25. Yoneyama T, Doi H, Kobayashi E, Hamanaka H. Effect of heat treatment with the mould on the super-elastic property of Ti-Ni alloy castings for dental application. *J Mater Sci Mater Med* 2002;13:947—51.
26. Miyazaki S, Ohmi Y, Otsuka K, Suzuki Y. Characteristics of deformation and transformation pseudoelasticity in Ti-Ni alloys. *J Phys Colloq* 1982;43. C4-255—60.
27. Thompson SA. An overview of nickel—titanium alloys used in dentistry. *Int Endod J* 2000;33:297—310.
28. Shen Y, Zhou HM, Zheng YF, Peng B, Haapasalo M. Current challenges and concepts of the thermomechanical treatment of nickel-titanium instruments. *J Endod* 2013;39:163—72.
29. Elsewify T, Elhalabi H, Eid B. Dynamic cyclic fatigue and differential scanning calorimetry analysis of R-Motion. *Int Dent J* 2023 (in press).
30. Arias A, Macorra JC, Govindjee S, Peters OA. Correlation between temperature-dependent fatigue resistance and differential scanning calorimetry analysis for 2 contemporary rotary instruments. *J Endod* 2018;44:630—4.
31. Shim KS, Oh S, Kum K, Kim YC, Jee KK, Chang SW. Mechanical and metallurgical properties of various nickel-titanium rotary instruments. *BioMed Res Int* 2017;2017:4528601.
32. Capar ID, Ertas H, Arslan H. Comparison of cyclic fatigue resistance of nickel-titanium coronal flaring instruments. *J Endod* 2014;40:1182—5.
33. Grande NM, Plotino G, Silla E, et al. Environmental temperature drastically affects flexural fatigue resistance of nickel-titanium rotary files. *J Endod* 2017;43:1157—60.
34. 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—6.
35. Dosanjh A, Paurazas S, Askar M. The effect of temperature on cyclic fatigue of nickel-titanium rotary endodontic instruments. *J Endod* 2017;43:823—6.
36. Unno H, Ebihara A, Hirano K, et al. Mechanical properties and root canal shaping ability of a nickel-titanium rotary system for minimally invasive endodontic treatment: a comparative in vitro study. *Materials* 2022;15:7929.
37. Nishijo M, Ebihara A, Tokita D, Doi H, Hanawa T, Okiji T. Evaluation of selected mechanical properties of NiTi rotary glide path files manufactured from controlled memory wires. *Dent Mater J* 2018;37:549—54.
38. Tokita D, Ebihara A, Miyara K, Okiji T. Dynamic torsional and cyclic fracture behavior of ProFile rotary instruments at continuous or reciprocating rotation as visualized with high-speed digital video imaging. *J Endod* 2017;43:1337—42.
39. Kaval ME, Capar ID, 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—3.
40. Kim W, Oh S, Ryu GJ, al at. Effect of autoclave sterilization on cyclic fatigue and torsional fracture resistance of NiTi rotary instruments. *Odontology* 2020;108:194—201.
41. Plotino G, Grande NM, Cordaro M, Testarelli L, Gambarini G. A review of cyclic fatigue testing of nickel-titanium rotary instruments. *J Endod* 2009;35:1469—76.