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Significance of heat treatment on the mechanical and fatigue properties of NiTi endodontic rotary files

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ABSTRACT

The present investigation is conducted to address the relationship between microstructure and phase transformation with functional properties of NiTi rotary files. For this purpose, heat treatment at 460–500 °C for 90–120 min followed by cooling in water, air and furnace was accomplished on rotary files fabricated by grinding of as-drawn wires with a chemical composition of Ni-49.2 at.% Ti. Prior to heat treatment, the rotary files exhibited an austenitic phase and demonstrated complete superelasticity during bending tests. Heat treatment resulted in a multiphase microstructure (B19', R, and B2 phases) exhibiting shape memory effect together with superelasticity and therefore, enhancing the flexibility of rotary files. The heat treatment at 460 °C for 120 min followed by furnace cooling revealed the maximum hardness value of ~350 Hv and the best fatigue resistance with a number of cycles to fracture (NCF) of ~1000 during rotation bending fatigue test in a simulated root canal. It was suggested that microstructural and phase engineering is feasible by controlling the heat treatment variables to find remarkable functional properties to produce high-performance endodontic rotary files.

1. Introduction

Nickel-titanium (NiTi) alloys as smart materials exhibit unique functional properties such as superelasticity (SE) and shape memory effect (SME), making them invaluable in medical and engineering applications [1–3]. Thermoelastic martensitic phase transformations from high-temperature austenite (B2 phase) to the low-temperature martensite (B19' phase) is responsible for occurrence of SE and SME in NiTi alloys. These recoverable strains, achieved through unloading (SE) or heating (SME), are determined by phase transformation temperatures which depend on alloy composition, thermal processing and application conditions [4,5]. Preventing slip mechanisms activation, which leads to permanent deformation during straining, is the key role in order to obtain superior SE and SME. Microstructure tuning by grain refinement, strain hardening and solid solution strengthening together with precipitation-hardening, by formation of Ni_4Ti_3 in Ni-rich NiTi alloy, enables to improve the strength of the materilas and therefore, provides SE and SME [6,7]. It is well-established that perfect SE and SME effects are not expected in the NiTi alloy in as-cast, solution annealed (fully annealed) or cold-rolled conditions. Thus, heat treatment following solution annealing or cold rolling is essential to develop a desirable microstructure with adequate strength, minimizing permanent deformation and achieving superior SE and SME [8–10]. Thermomechanical treatment, involving controlled plastic deformation followed by heat treatment, is an effective method to tailor the microstructure and enhance both structural and functional properties, such as SE and SME. Thermomechanical treatments significantly influence phase transformation behaviour, including transformation temperatures, thermal hysteresis and the promotion of two-step phase transformation leading to intermediate R-phase formation. These changes directly impact the mechanical properties and performance of NiTi alloys in cyclic applications [4,5].

In addition to the noted unique properties of SE and SME, NiTi alloys are distinguished by their excellent biocompatibility and corrosion resistance. These characteristics make NiTi alloys in dispensable for a wide range of medical applications, including sensors, actuators, connection devices, cardiovascular stents, orthodontic wires and endodontic rotary files [1]. Endodontic treatment, involving the removal of inflamed or necrotic pulp tissue and canal filling, is challenging due to the complex root canal anatomy [11–13]. In the 1990s, NiTi alloys were introduced to improve flexibility, with the first NiTi rotary file released in 1993 [14]. In the early 2000s, heat treatment was found to enhance

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the flexibility of NiTi files by modifying their transformation behavior [15–18]. The martensitic phase of NiTi offers exceptional fatigue resistance, allowing instruments to deform and recover their shape when heated to the austenitic phase, making them superelastic. Proprietary thermomechanical treatments integrate hardening and heat treatment into one process, enhancing material properties. These advancements aim to preserve the structure of alloy for optimal strength, as opposed to traditional grinding methods that can cause microfractures [19]. M-wire and R-phase treated NiTi alloys, which offer enhanced flexibility and fatigue resistance, represented the next generation of rotary files [20–23]. Studies also show that these alloys improve centering ability and reduce canal transportation [24]. It is important to note that, autoclaving does not significantly affect the flexibility or fracture resistance of these advanced NiTi files, even after multiple sterilization cycles [25].

As noted before, the extraordinary properties of the NiTi alloys provide the ability to tolerate high bending and torsional stresses during root canal treatment, enhancing treatment efficiency and reducing the risk of fracture during rotation. Basically, this reliability is crucial for ensuring successful and safe root canal procedures. The efficiency of rotary files depends significantly on their fatigue resistance during cyclic straining in the root canal. The fatigue life of NiTi alloys is strongly influenced by transformation temperatures and cyclic loading conditions [26]. For instance, higher transformation temperatures can enhance fatigue resistance by promoting martensitic phase stability during cyclic loading [26,27].

It is well established that the martensitic phase, being softer and more flexible than the austenitic phase, provides superior fatigue properties, particularly in alloys containing B19' and R phases [4,5,28]. Superelastic rotary files have demonstrated limitations in highly curved canals due to their stiffness and reduced fatigue resistance [29,30]. In fact, phase transformation behavior can be tailored through heat treatment parameters, which significantly influence the performance and fatigue life of the alloy [26,31]. Parameters such as heat treatment temperature and time together with cooling rate are critical in optimizing phase transformation and mechanical properties in NiTi alloys by enhancing flexibility and fatigue resistance [4,32].

There are numerous reports on the effects of chemical composition, machining parameters and rotary file design on the performance of rotary files [10,26,27,31,33]. However, limited information is available on the effect of microstructure engineering achieved through controlled heat treatment following plastic deformation on the functional and fatigue properties of rotary files. Understanding this relationship is crucial for optimizing performance in clinical applications. Accordingly, this investigation aims to explore the relationship between microstructure and phase transformation and their impact on mechanical behavior, including SME and SE effects, as well as fatigue properties in NiTi rotary files. This study seeks to establish how controlled heat treatments can enhance fatigue resistance and flexibility in rotary files. For this purpose, heat treatment under various conditions was accomplished on rotary files fabricated from as-drawn wires. This study represents a step forward to understand the behavior of the smart NiTi alloys with complex microstructure to produce high performance endodontic rotary files.

2. Materials and methods

This investigation was conducted on endodontic rotary files fabricated by Hamerz Medical Co. These rotary files were fabricated by grinding of as-drawn NiTi wires with a diameter of 1.2 mm. The chemical composition of the studied alloy wasNi-49.2Ti (at. %). The surface topography and cross-sectional area of the endodontic rotary files were examined using a field emission scanning electron microscope (FE-SEM, TESCAN MIRA3) equipped with an energy-dispersive X-ray spectrometer (EDS) and optical microscopy (OM). For microstructural investigation, the cross-sections of the samples were mechanically polished and etched using aH_2O:HNO_3:HF (4.5:3:1.5) solution. Heat treatment on the fabricated endodontic rotary files was conducted at 400, 420, 440, 460, 480 and 500 °C for 90, 100 and 120 min followed by water quenching (WQ), air cooling (AC) and furnace cooling (FC). Bending behavior of heat-treated samples were studied based on the procedure and special fixture design reported earlier [34]. It is important to note that the magnitude of applied strain was 3% and the samples were heated up to 70 °C after unloading to measure the recovered strain by heating.

Phase transformation behaviour of samples before and after heat treatment was studied by differential scanning calorimetry (DSC) using a Mettler-822e DSC instrument with a liquid nitrogen cooling. The heating and cooling regimen was as follows: specimens were heated up quickly to 150 °C and kept at this temperature for 5 min followed by cooling down to −100 °C in order to achieve the corresponding cooling curve. After keeping at −100 °C for 5 min, the sample heated up to 150 °C in order to achieve the corresponding heating curve. It is important to note that cooling and heating rates during thermal cycling were 10 °C.min^{-1}. The transformation temperatures were determined by the extrapolated tangential method. X-ray diffraction (XRD, X'Pert MPD model) was used to study the phases before and after heat treatment employing Cu Kα radiation (wavelength $\lambda = 0.154$ nm) at 40 kV and a tube current of 40 mA. XRD measurements were carried out over an angular 2θ range from 35° to 55° using a scanning step of 0.01° and a scanning speed of 2 deg.min^{-1}. The percentage of phases in the samples was calculated based on equation suggested earlier [35].

Longitudinal sections of rotary files were polished to a mirror-like quality and hardness measurements were taken using a Vickers microhardness tester with a load of 100 gf and adwell time of 10 s. Every point the local value of Hv was obtained from theaverage of at least three separate hardness values. Cyclic rotating bending fatigue test on heat treated rotary files was carried out by operating therotary instrument with a handpiece (E-Connect manufactured by Eighteeth Company) in a simulated root canal consisting in a steel block with matching curvature of 5 mm (Fig. 1). This setup replicates clinical conditions encountered in curved root canals, providing insights into the rotary file's performance under realistic mechanical stresses. The rotational speed was fixed at 300 rpm and the handpiece allowed momentary torque recording (0.1 s intervals) with a sensitivity of 0.05 N cm. At least ten samples were tested to failure for each condition. It is important to note that the specimens were video recorded during testing, providing an accurate estimation of the number of cycles to failure. The fracture surfaces of the samples were analyzed using the FE-SEM (TESCAN MIRA3).

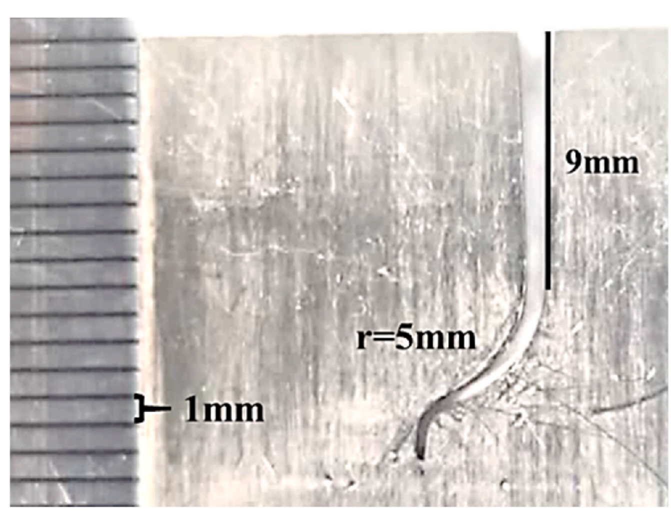

Fig. 1. Simulated root canal in a steel block used for cyclic rotary bending fatigue tests, illustrating a curvature radius of 5 mm.

3. Results

Fig. 2a and b presents surface topography and longitudinal cross section of a rotary file after grinding analyzed by SEM and OM, respectively. The surface topography of the samples reveales high-quality surface, characterized by relatively smooth flute areas and the absence of machining chips on the cutting edges. This smooth surface is critical for reducing stress concentration points, thereby enhancing fatigue resistance during clinical use. Fig. 2b shows the distribution of minor Ti_4Ni_2O oxide particles (<2%) with an average size of ~1 μm within the microstructure of the NiTi alloy, as identified by energy-dispersive X-ray spectroscopy (EDS) analysis. These oxide particles may influence mechanical performance by acting as sites for crack initiation under cyclic loading. The presence of these oxide particles has been previously observed and documented in NiTi alloys [20,21]. This observation aligns with earlier reports, which also highlighted the role of Ti_4Ni_2O particles in influencing the fatigue resistance and phase stability of NiTi alloys.

Fig. 3 illustrates bar charts showing the recovered bending strain after loading and unloading (E + SE), as well as heating up to 70 °C (SME) for NiTi rotary files before and after heat treatment under various conditions followed by air cooling. This analysis highlights the distinct contributions of superelasticity (E + SE) and shape memory effect (SME) to the mechanical behavior of the rotary files. The measured recovered strain values were summarized in Table 1 after heat treatment of NiTi rotary files at temperatures ranging from 400 to 500 °C in 20 °C increments with durations of 90, 100 and 120 min followed by water quenching (WQ), air cooling (AC) and furnace cooling (FC). The results indicate that the untreated rotary files (as-drawn material) exhibited no SME. However, heat treatment leads to SE together with SME in which the portion of the SE is remarkable in the samples. This demonstrates that the microstructural changes induced by heat treatment are essential for enabling shape memory and superelastic behavior. Detailed analysis suggests that SME increases with higher temperatures and longer durations of heat treatment. Furthermore, slow furnace cooling resulted in higher SME compared to faster cooling methods such as water quenching and air cooling. This effect may be attributed to the enhanced stabilization of the R-phase and martensite during gradual cooling, as confirmed by DSC and XRD results. The heat-treated sample at 500 °C for 120 min followed by furnace cooling exhibited the highest SME (~1.5%). This optimized heat treatment condition may be particularly suitable for applications requiring high flexibility and fatigue resistance in rotary files.

Fig. 4 illustrates the DSC curves of rotary files after heat treatment at 460, 480 and 500 °C for 120 min followed by furnace cooling and transformation temperatures were summarized in Table 2. It is important to note that these samples exhibited significant SME, as detailed in Table 1. The appearance of exothermic peaks during cooling and endothermic peaks during heating confirms the occurrence of austenite-to-martensite transformation and its reverse in the samples. The start, peak and finish transformation temperatures are denoted by subscripts *s*, *p*, and *f*, respectively, while ΔT represents the temperature difference between forward and reverse transformations. The DSC results display two broad peaks corresponding to the transformation from austenite to the R phase and then the R phase to martensite transformation upon cooling and the two merged peaks correspond to the reverse transformation of martensite to the R phase and the R phase to austenite upon heating. It is important to note that only a single endothermic peak was observed for the heat-treated sample at 500 °C. The results indicate that phase transformations become more pronounced, and the peaks tend to merge as the heat treatment temperature increases. The transformation temperatures, including Rs (29–46 °C) and Mf (−4 to −25 °C) upon cooling and As (24–39 °C) upon heating suggest that the structure after heat treatment and furnace cooling to room temperature contains a mixture of R-phase, martensite and austenite in the samples.

The X-ray diffraction patterns of samples before and after heat treatment at 460, 480 and 500 °C for 120 min followed by furnace

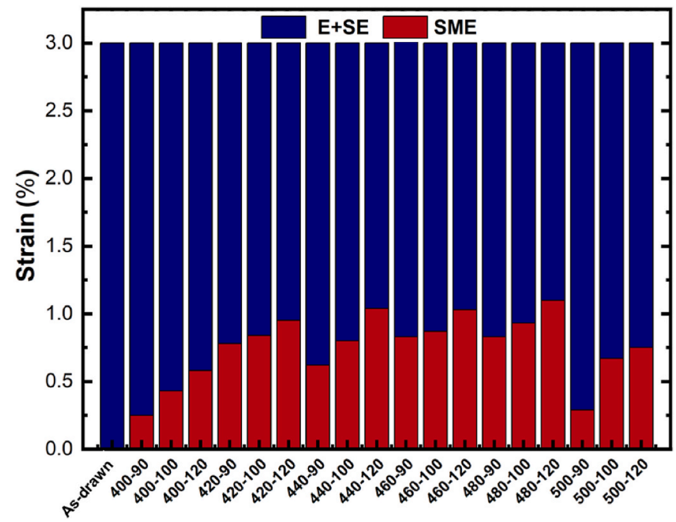

Fig. 3. Recovered bending strain (E + SE and SME) of NiTi rotary files before and after heat treatment at 400, 420, 440, 460, 480 and 500 °C for 90, 100, and 120 min followed by air cooling.

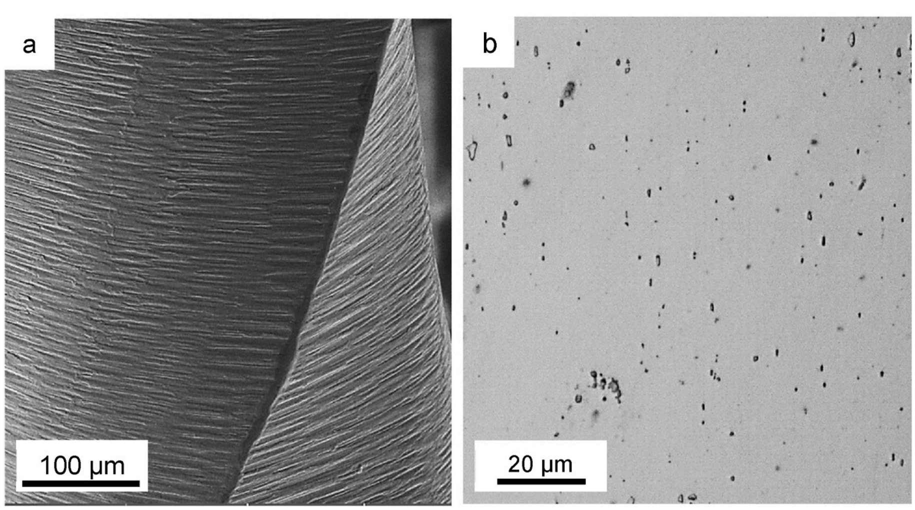

Fig. 2. (a) SEM micrograph showing the surface topography and (b) optical microscopy image of the longitudinal cross-section of the rotary file.

Table 1
Bending properties (E + SE and SME) of NiTi rotary files after heat treatment at 400, 420, 440, 460, 480 and 500 °C for 90, 100 and 120 min followed by water quenching (WQ), air cooling (AC) and furnace cooling (FC).

Heat treatment conditions			Bendig propertiers	
Temperature (°C)	time (min)	Cooling condition	E + SE (%)	SME (%)
400	90	AC	2.75 ± 0.01	0.25 ± 0.01
	100	AC	2.37 ± 0.35	1.63 ± 0.35
	120	AC	1.87 ± 0.01	1.13 ± 0.01
420	90	AC	2.22 ± 0.32	0.78 ± 0.32
	100	AC	2.16 ± 0.04	0.84 ± 0.04
	120	AC	2.05 ± 0.28	0.95 ± 0.28
440	90	AC	2.38 ± 0.03	0.62 ± 0.03
	100	AC	2.20 ± 0.02	0.80 ± 0.02
	120	AC	1.96 ± 0.08	1.04 ± 0.08
460	90	AC	2.18 ± 0.08	0.83 ± 0.08
	100	AC	2.13 ± 0.03	0.87 ± 0.03
	120	AC	1.97 ± 0.02	1.03 ± 0.02
	120	FC	1.83 ± 0/01	1.18 ± 0.01
480	90	AC	2.07 ± 0.08	0.93 ± 0.08
	100	AC	2.17 ± 0.07	0.83 ± 0.07
	120	AC	1.90 ± 0.02	1.10 ± 0.02
	120	WQ	2.40 ± 0.00	0.60 ± 0.00
	120	FC	1.74 ± 0.01	1.26 ± 0.01
500	90	AC	2.71 ± 0.06	0.29 ± 0.06
	100	AC	2.33 ± 0.03	0.67 ± 0.03
	120	AC	2.25 ± 0.05	0.75 ± 0.05
	120	FC	1.54 ± 0.01	1.46 ± 0.01

cooling are shown in Fig. 5. The microstructure before heat treatment is fully austenite which supports the SE behaviour observed during bending test. Heat treatment leads to the appearance of new peaks corresponding to R and B19' martensitic phases which supports the DSC results and explains SME of these samples during the bending test. The results show that the amount of R-phase and austenite increases and decreases, respectively, by increasing the temperature of heat treatment. The calculated values of the R-phase from XRD results were 42 and 60% for the temperature of 460 and 500 °C, respectively.

The microhardness measurements of NiTi rotary files after heat treatment at 460, 480 and 500 °C for 120 min followed by furnace cooling are shown in Fig. 6a. Close inspection reveals there is a decrease in the hardness from ~350 to ~310 Hv with increasing the temperature of heat treatment from 460 to 500 °C. It is important to note that the microhardness value for the sample before heat treatment (as-drawn) was ~260 Hv. It suggests formation of precipitates and therefore activation of precipitation strengthening mechanism in the heat-treated samples. Fig. 6b illustrates the number of cycles to fracture (NCF) of heat-treated rotary files after rotary bending tests. This value was measured as ~999, 995 and 770 NCF for heat-treated samples at 460, 480 and 500 °C, respectively, which confirm a significant drop in fatigue resistance by increasing the temperature from 480 to 500 °C. The results reveal very close properties for heat-treated samples at 460 and 480 °C.

Fig. 7 illustrates a set of SEM micrographs of fracture surfaces after rotary bending tests in different magnifications. Areas marked by 2 and 3 represent crack propagation and final fracture areas, respectively. The microstructural observations clearly show the characteristics of rotary bending fatigue fracture under high level of loading [27]. There are many positions of crack initiations on the cutting edges and flute areas linked to brittle features related to fatigue crack propagation. The final fracture with ductal features is located at the center of the fracture surface.

4. Discussion

The phase transformations in NiTi alloys are highly sensitive to heat tereatment conditions. The results clearly show the effect of heat treatment variables on the microstructure and functional properties of NiTi endodontic rotary files. The sample before heat treatment revealed only SE behavior, with no detectable SME. Structural analysis confirmed a stable autistic phase in the as-drawn sample. A stable austenitic phase was predictable in this condition due to the chemical composition of the alloy which was considered as Ni-rich NiTi alloys (Ni-48.2 at.% Ti) with age hardening potential. It was suggested that heat treatment of these alloys at moderate temperatures (300–500 °C) leads to formation of very fine lenticular-shaped Ni_4Ti_3 precipitates which improves the strength

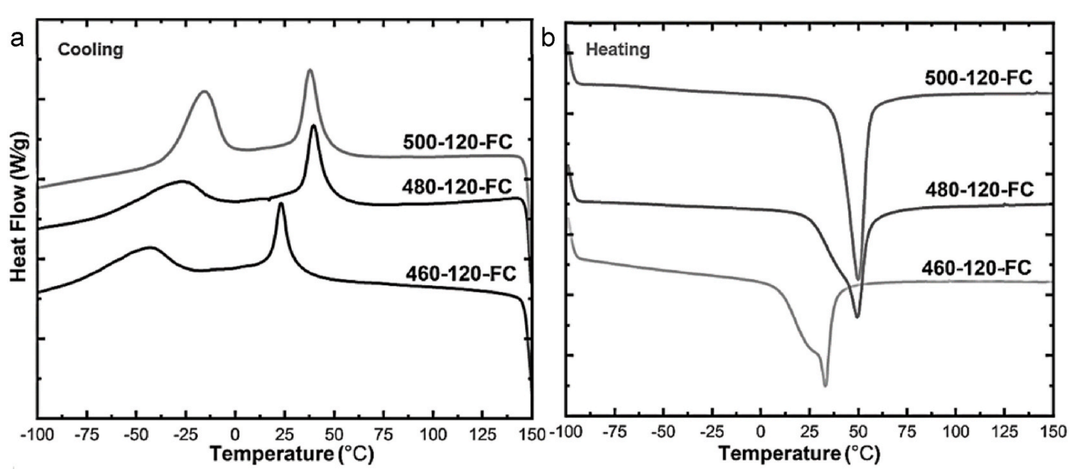

Fig. 4. DSC curves at (a) cooling and (b) heating regims for heat treated samples at 460, 480 and 500 °C for 120 min followed by furnace cooling.

Table 2
Phase transformation temperatures for NiTi rotary files heat-treated at 460, 480 and 500 °C for 120 min with furnace cooling, showing the start (s), peak (p) and finish (f) temperatures for forward and reverse transformations, as well as the transformation temperature difference (ΔT).

Heat treatment	Phase transformation temperatures (°C)									
	R_S	R_P	R_f	M_s	M_p	M_f	A_s	A_p	A_f	ΔT
460-120-AC	29	23	17	−25	−43	−70	24	33	37	76
480-120-FC	46	39	33	−10	−27	−59	37	49	55	76
500-120-FC	45	37	31	−4	−16	−33	39	49	55	65

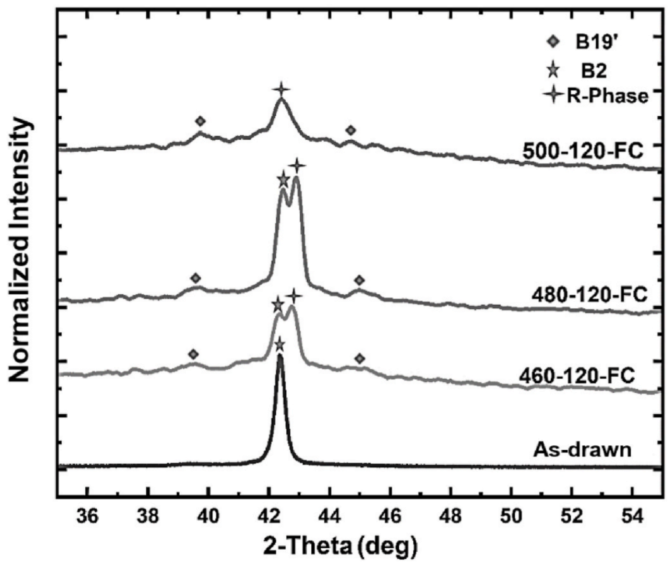

Fig. 5. X-ray diffraction patterns of NiTi rotary files before and after heat treatment at 460, 480 and 500 °C for 120 min followed by furnace cooling.

of the alloy and depletes the Ni from the matrix simultaneously [6,7]. The former provides better functional properties because it prevents activation of slip as permanent deformation and the latter leads to decreased transformation temperature in the alloy [6,36–38]. The microhardness increment from ~260 to ~350 Hv after conducted heat treatment can be considered as indirect evidence of precipitation hardening in the studied samples. It is readily confirmed that any deviation from the equiatomic NiTi to the Ni-rich side of phase diagram leads to decrease phase transformation temperatures significantly [35]. Accordingly, depletion of the alloy matrix from Ni increases the phase transformation and encourages formation of martensitic phase. Increasing the phase transformation observed in DSC results (Fig. 4) and the appearance of martensitic phases in XRD patterns (Fig. 5) suggest the occurrence of precipitation in the microstructure of the studied samples. The DSC analyses revealed two well-separated peaks during cooling, corresponding to the transformations from austenite to R-phase and subsequently to martensite. It was reported earlier that the strain fields of Ni_4Ti_3 precipitates with coherent interface with the matrix, encourage R-phase transformation in NiTi alloys. In fact, lattice distortion and volume change associated with R-phase formation are lower than those of the B19' martensitic phase adjacent to formed Ni_4Ti_3 precipitates formed during heat treatment [6,7]. Thus, the presence of SME in the heat-treated samples and its enhancement with increased heat treatment temperature and time are consistent with these findings. It is important to note that the formation of stable martensitic phases together with the austenite provides more flexibility in the endodontic rotary file [34].

It is worth noting that increasing the heat treatment temperature or time may increase transformation temperature and lead to appearance SME in the alloy, nevertheless, it may deteriorate the mechanical properties of the alloy which may affect functional properties including SME and SE. In fact, activation of softening mechanisms such as recovery and grain growth together with the coarsening of Ni_4Ti_3 precipitates during prolonged term heat treatment at elevated temperatures lead to decrease the strength of the alloy and occurrence of plastic deformation as an unrecoverable process [6,39–41]. Consequently, higher heat treatment temperatures or extended durations were intentionally avoided in this study. The results indicate a significant decrease in sample hardness with increasing heat treatment temperature,

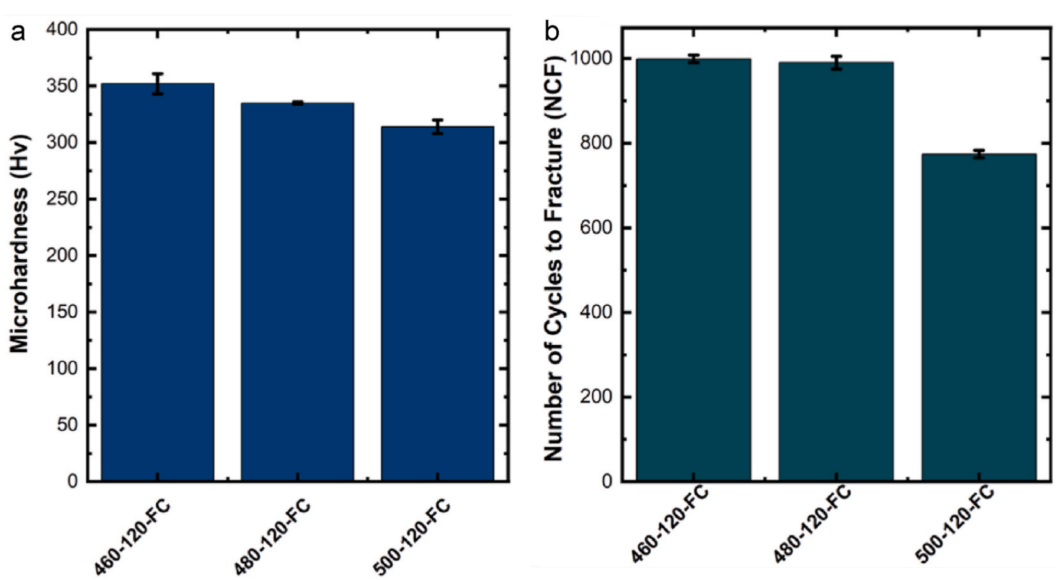

Fig. 6. a) Vickers microhardness values and b) number of cycles to fracture (NCF) after rotary bending tests of heat-treated NiTi rotary files at 460, 480, and 500 °C for 120 min.

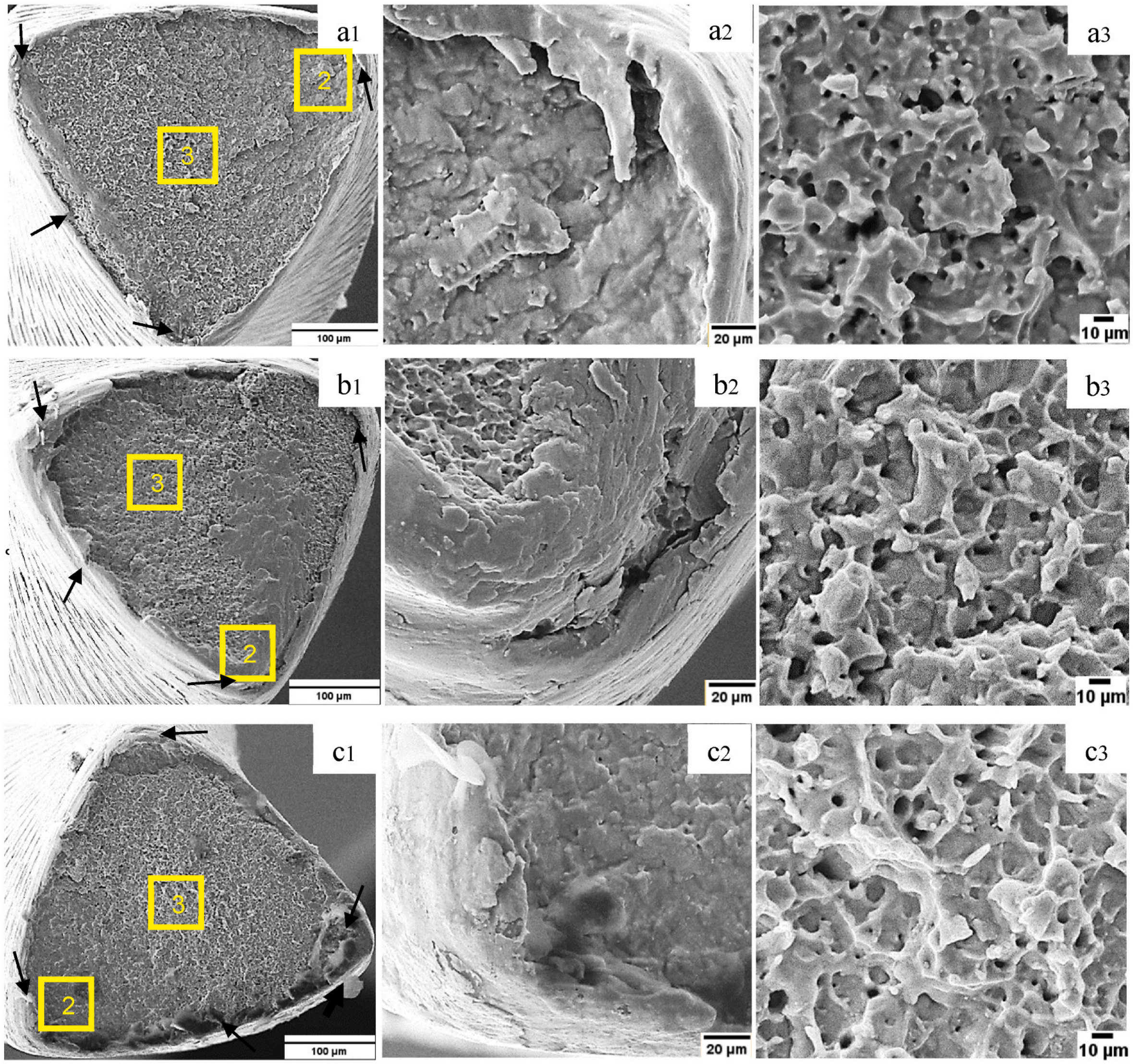

Fig. 7. SEM micrographs of fracture surfaces of heat-treated NiTi rotary files at a) 460, b) 480 and c) 500 °C for 120 min. Black arrows show crack initiation sites, and areas labeled by 2 and 3 represent crack propagation and final fracture areas, respectively.

dropping from ~350 to ~310 Hv as the temperature increased by only 40 °C. This reduction in hardness can be attributed to the coarsening of precipitates and the initiation of grain recovery processes. It was shown earlier that the recrystallization temperature of the alloy is around 500 °C [32,34,41–44].

Rotating files within the dental canal experience high levels of bending and torsional stress, which rapidly initiate microcracks at various surface locations [45]. A defect-free, well-polished surface is crucial for significantly improving the fatigue properties of rotary files [46]. Surface conditions, shape design, and the alloy's microstructure are critical parameters influencing the fatigue life of rotary files. Detailed examination of the rotary file surface (Fig. 2a) reveals fine polishing, which likely delays crack initiation. Microstructural analysis confirms a low volume fraction of homogeneously distributed fine oxides. It is noteworthy that brittle particles with weak interfaces act as crack initiation sites, particularly in high-cycle fatigue, where crack initiation predominantly governs fatigue life. In low-cycle fatigue, cracks are expected to initiate at the surface during the early stages of cyclic deformation, with crack growth predominantly controlling fatigue life [47,48]. Accordingly, smaller particle sizes with lower volume fractions are associated with improved mechanical properties [49,50]. The fine oxides observed in the current study likely contribute to the enhanced fatigue resistance of the heat-treated files. The results indicate that the oxide volume fraction is within acceptable limits (<5%) [51]. This ensures minimal adverse effects on mechanical performance while maintaining surface integrity.

It is well-established that increasing the hardness and strength of alloys enhances their fatigue properties [52]. Fig. 6 illustrates a correlation between hardness values and the NCF for heat-treated samples. The structural and microstructural characteristics of the alloy are critical in controlling the crack propagation stage, which dominates low-cycle fatigue behavior. The formation of a multiphase structure in NiTi alloys has been shown to improve fatigue properties compared to those of single-phase austenitic alloys [53]. Furthermore, evidence supports the benefits of optimized heat treatment in tailoring microstructures to strengthen alloys and enhance low-cycle fatigue resistance [54,55]. The present results show that rotary file heat-treated at 460 and 480 °C exhibit convincing fatigue resistance. This performance can be attributed to their multiphase structure, the formation of a high volume fraction of martensitic phase, microstructures with residual defects from pre-heat treatment plastic deformation and formation of fine precipitates during heat treatment, all of which enhance the strength. These factors collectively reduce crack propagation rates, thereby extending the fatigue life of the rotary files. Notably, rotary files heat-treated at

460 and 480 °C exhibited similar phases, microstructures and hardness resulting in comparable fatigue properties. However, increasing the heat treatment temperature from 480 to 500 °C resulted in deterioration of fatigue properties. It can be related to the formation of an almost single-phase microstructure and a reduction in microhardness due to recovery, recrystallization and the coarsening of Ni_4Ti_3 precipitates. These findings confirm the importance of maintaining heat treatment temperatures below 500 °C to preserve the desired balance between hardness and fatigue resistance.

Table 3 summarizes the results of fatigue tests conducted on different NiTi endodontic rotary files under similar conditions as those investigated in the present study [55–63]. A detailed examination of the results reveals that the heat-treated rotary files in this study exhibit superior fatigue properties compared to commercially available NiTi endodontic rotary files. The superior performance of heat-treated files can be attributed to their optimized microstructure including a higher volume fraction of martensitic phases and improved phase transformation behavior, as demonstrated in XRD and DSC analyses. It is important to highlight that while fatigue resistance is a critical parameter, it is not the sole determinant of the performance and efficiency of rotary files.

5. Conclusion

Microstructural and phase engineering is feasible by controlling the heat treatment parameters on a deformed NiTi alloy in order to find remarkable functional properties in endodontic rotary files. Here are the main achievements of the current investigation:

1. Initial (as-drawn) condition of the rotary file represented an austenitic phase and therefore superelasticity (SE) under bending test. Surface topography analysis revealed a finely polished surface, while microstructural investigations confirmed the presence of a very low volume fraction (<2%) of fine oxides (~1 μm).
2. Heat treatment at 460–500 °C for 90–120 min followed by cooling in water, air and furnace led to occurrence of shape memory effect (SME) up to 50% of bending strain in the rotary files. DSC and XRD results suggested occurrence of martensitic transformation during cooling which provides a multiphase microstructure containing B19', R and B2 phases. The volume fraction of martensitic phases increased by increasing heat treatment temperature and time which provides better SME and therefore, more flexibility in the endodontic rotary file.
3. The hardness measurements and phase transformation temperatures proposed formation of Ni-rich precipitates during heat treatment of the studied Ni-rich alloy (Ni-49.2 at.% Ti). The depletion of Ni from the matrix during heat treatment likely increased phase transformation temperatures with higher heat treatment conditions. The microhardness values of the rotary files improved significantly from ~260 Hv in the as-drawn state to ~350, ~335, and ~310 Hv after heat treatment at 460, 480, and 500 °C, respectively, for 120 min, followed by furnace cooling. This hardness increment contributes to improved resistance against plastic deformation during cyclic loading.
4. Rotary files heat-treated at 460 °C for 120 min exhibited exceptional fatigue resistance, achieving a number of cycles to fracture (NCF) of ~1000 in rotary bending fatigue tests conducted in a simulated root canal. It can be due to their multiphase structure and the formation of a high volume fraction of martensitic phases, microstructures containing residual defects remained from pre-heat treatment plastic deformation and fine precipitates formed during heat treatment. However, increasing the heat treatment temperature to 500 °C resulted in a decline in fatigue resistance, likely due to the formation of an almost single-phase martensitic microstructure and reduced microhardness. This highlights the critical importance of optimizing heat treatment temperatures to balance microstructural benefits and mechanical performance.

Table 3
Fatigue resistance of NiTi endodontic rotary files tested under comparable conditions.

Rotary files	Canal Curvature Angle (°)	Rotation speed (rpm)	NCF	Ref
AF Rotary	90	500	605	[56]
WaveOne MT	45	300	550	[55]
ER	90	500	861	[57]
Reciproc Convén	60	300	844	[58]
Protaper Gold	60	300	739	[58]
TRUshape	60	300	768	[58]
Mani silk	45	500	626	[59]
R25(Group A4)	60	300	806	[60]
i3G-f	60	350	379	[61]
MiniScope	60	350	454	[62]
Neoendo Flex	60	400	490	[63]
Edgeendo	60	300	417	[63]
Denco	60	300	435	[63]
460-120-FC	45	300	999	This study
480-120-FC	45	300	995	
500-120-FC	45	300	770	

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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