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Effect of heat treatment on cyclic fatigue resistance, thermal behavior and microstructures of K3 NiTi rotary instruments

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Abstract

Objective. The aim of this study was to investigate the effect of heat treatment on the cyclic fatigue resistance, thermal behavior and microstructural changes of K3 NiTi rotary instruments. ***Materials and methods.*** Twelve control (as-received) and 12 experimental (heat-treated) K3 NiTi rotary instruments were compared in this study. Those experimental K3 instruments were heated in a furnace for 30 min at 450°C and then quenched in water. The cyclic fatigue resistance was measured with a fatigue tester. The thermal characteristic and the microstructures of both instruments were investigated by differential scanning calorimetry (DSC) and transmission electron microscopy (TEM), respectively. ***Results.*** There was a significant increase in the cyclic fatigue resistance between the heat-treated instruments and the as-received instruments (T-test, $p < 0.05$). DSC showed that the as-received and heat-treated samples were different, with an increased A_f (austenite-finish temperature) for the latter. TEM analysis revealed that both as-received and heat-treated instruments were composed mainly of an austenite phase. However, the heat-treated samples had an increased appearance of larger grains, twinning martensite, TiO_2 surface layer and a Ni-rich inner layer. ***Conclusions.*** Heat treatment increased the cyclic fatigue resistance of NiTi files and changed the thermal behavior of the instruments without marked changes in the constituting phases of NiTi alloy.

Key Words: *cyclic fatigue resistance, heat treatment, K3 NiTi rotary instrument, microstructural changes, thermal behavior*

Introduction

In contemporary endodontics, clinicians use engine-driven Nickel-Titanium (NiTi) rotary instruments to make the endodontic procedure more efficient [1]. However, accidental separation of NiTi rotary instruments is a concern. The two main mechanisms of NiTi rotary instrument fracture were reported to be responsible: cyclic fatigue fracture and torsional fracture [2]. Factors which affect the cyclic fatigue resistance of NiTi rotary instruments included (micro) structures of NiTi alloy [3], corrosion [4], root canal geometry [5], instrumentation motion [6,7], cross-sectional configuration [8] and surface topography [9,10]. Metallurgically, annealing and heat treatments are possible means of altering the mechanical properties of NiTi alloys [11,12]. The mechanical properties of the NiTi alloy are also affected by chemical composition, especially the nickel content of the alloy [13]. Recently, thermal treatment of the NiTi alloy has been used to optimize the mechanical properties of NiTi rotary instruments [14,15]. Studies demonstrated that the heat treatment improved the bending property and flexibility [16,17] and cyclic fatigue resistance [17–21]. Although these studies showed that heat treatment is able to improve the mechanical

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properties of NiTi rotary instruments, they failed to provide information regarding the underlying mechanism for such improvements. Knowledge on the constituting phases, grain size or chemical composition of the heat-treated instruments is lacking.

Conventional NiTi rotary files are produced by a grinding process. During this procedure, a considerable amount of work-hardening takes place on the surface; machining grooves or marks are also produced there. Heat treatment, that is heating of the material and subsequent quenching, is a widely known way of stress-releasing work-hardened materials. Depending on the holding temperature and time and the rate of cooling for the hot material, the microstructure (including grain size or any phase separation) of the NiTi alloy may occur [22]. Heat treatment can lead to alteration of the NiTi lattice and its phase transformation characteristic [23]. The effect may be confirmed by changes in the transition temperatures (thermal behavior). In fact, differential scanning calorimetry (DSC) analysis is a method to compare the thermal behavior of NiTi rotary instruments [24]. On the other hand, the microtextures and grain size cannot be confirmed by DSC. Transmission electron microscopy (TEM) or X-ray diffraction analysis is necessary to examine the crystal lattice [25]. In fact, a selected area diffraction pattern (SADP) accompanied with TEM is the most precise tool for identifying the constituent phases of NiTi alloy. Considering that there is a lack of knowledge to explain the metallurgical changes induced in a NiTi rotary instrument by heat treatment, it is quite necessary to investigate the thermal behavior changes and microtexture changes induced by heat treatment with the help of recent technologies, such as DSC and TEM. Thus, the aim of this present study was to examine the effect of heat treatment on the cyclic fatigue resistance, thermal DSC behavior and microstructures of a K3 NiTi rotary instrument.

Materials and methods

Heat treatment

K3 NiTi rotary instruments (SybronEndo, Orange, CA) of size 25 and 0.06 taper were used in this experiment. The heat treatment comprised heating in a furnace (Focus 2010, Shenpaz, Tel-Aviv, Israel) for 30 min at 450°C, followed by immediate quenching in water. The control group consisted of as-received K3 NiTi rotary instruments.

Cyclic fatigue resistance test

Cyclic fatigue resistance was measured for 12 as-received and 12 heat-treated K3 NiTi rotary instruments. All of them were inspected for any signs of deformation under a dental surgical microscope (OPMI, Zeiss, Oberkochen, Germany) before the test. Any deformed instrument was excluded. A fatigue tester (Denbotix, Bucheon, Korea) which allows pecking motions was used in this experiment. The instrument was mounted on an electric torque-controlled motor (Aseptico, Woodinville, WA) with a 20:1 reduction handpiece and rotated at the speed recommended by the manufacturer (300 rpm). Each file was set to run in an artificial canal of 1.5 mm diameter, with 60° curvature and 5 mm radius. A 6 mm pecking movement was applied at 0.5 cycles per second. During instrumentation, the artificial canal was filled with RC-prep (Premier Dental Products, Norristown, PA) to reduce the friction and to dissipate any heat generated. When the instrument fractured in the canal, the internal sensor within the tester detected the torque change. The motor then stopped and the time was automatically recorded digitally to the nearest of 0.1 s. The fracture was also visually confirmed. The time to fracture was multiplied by the rotation rate (rpm), to obtain the number of cycles to failure (NCF) for both the as-received and heat-treated group. Using the SPSS statistical package version 12 (SPSS, Chicago, IL), the NCF of the two groups were examined with *T*-test, at a significance level of $p = 0.05$.

Differential scanning calorimeter (DSC) analysis

The thermal behavior of the as-received and heat-treated K3 NiTi rotary instruments was measured with DSC (TA Instruments, New Castle, DE). Small segments of the files were placed in an aluminum pan on a Platinum holder inside the measuring chamber of DSC. An empty pan was included as a reference. The chamber was filled with high purity argon gas to minimize any oxidation. The exothermic or endothermic energy flow was recorded by raising the temperature to 100°C and subsequently cooled to −100°C and then re-heated to 100°C. The coolant used in this experiment was liquid nitrogen. The heating and cooling rate was 0.17°C/s. The M_s (martensite transformation starting point), M_f (martensite transformation finishing point), A_s (austenite transformation starting point) and A_f (austenite transformation finishing point) were determined.

Transmission electron microscopy

For TEM analysis, samples were prepared by dual beam focused ion beam (DB-FIB) (Helios Nanolab 600, FEI, Hillsboro, OR). The DB-FIB system has an ion column with a liquid ion source of Gallium (Ga+) for sectioning and a Schottky field emission electron column for imaging. Uniform and damage-free TEM samples were obtained with a thickness of less than 100 nm using variable accelerating voltages between 1–30 kV. Selected Area Diffraction Analysis (SADA) was also performed. The microstructure analysis

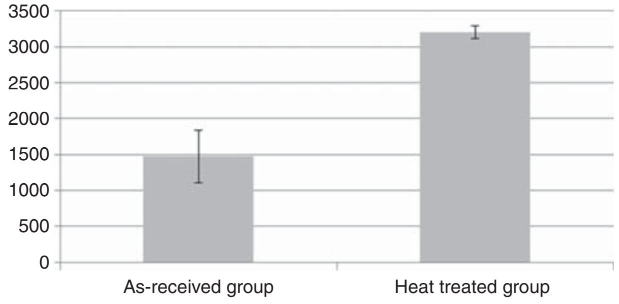

Figure 1. The result of cyclic fatigue resistance test (NCF: Number of Cycles to Failure).

was carried out by using a 200 kV TECNAI F20 G2 SuperTwin TEM (FEI, Hillsboro, OR) with the sample mounted in a double-tilt holder (Gatan, Pleasanton, CA). TEM images were obtained by UltraScan 1000 (2kX2k) CCD camera (Gatan Inc.) and Fischhione Model 3000 ADF detector. Qualitative and quantitative compositional analysis was done using energy dispersive spectroscopy (EDX PV9761, AMETEK, Inc. Berwyn, PA).

Results

For the fatigue test, the number of cycles to fracture (NCF) of heat-treated K3 NiTi rotary instruments (3200 ± 723) was significantly higher than that of as-received K3 NiTi instruments (1473 ± 185) ($p = 0.001$) (Figure 1). The DSC chart showed a single endothermic peak on heating (lower part of the curve in Figure 2A) of the as-received K3 NiTi rotary instruments with an onset temperature of −32.87°C, which corresponded to the beginning of martensitic to austenitic transformation; its austenite finishing temperature (A_f) was ~ 0°C. On cooling (upper curve in Figure 2A), the martensitic starting temperature was −6.63°C. For the heat-treated K3 NiTi rotary instruments (Figure 2B), two endothermic peaks occurred on heating, indicating transformation of the material via an intermediary R-phase. The onset temperatures for these two peaks are 10.89°C and ~ 27°C. The austenite finishing temperature (A_f) of heat-treated K3 NiTi rotary instrument was ~ 47°C. On the cooling curve, two exothermic peaks with an onset temperature of 25.11 and −53.75°C were seen, which also suggested a two-stage transformation from austenitic via the R-phase to martensitic NiTi.

Surface changes due to heat treatment

The results of TEM and elemental analysis of the surface of the as-received K3 NiTi instrument and heat-treated K3 NiTi instruments are shown in Figure 3. It was apparent that the NiTi composition varied according to the distance from the surface into the bulk of the material. For the as-received K3 NiTi rotary instruments, the inner zone (red square #1, Figure 3A) was almost entirely composed with nickel and titanium and contained almost no oxygen. The intermediate layer (red square #2, Figure 3A) contained oxygen, nickel and titanium with an atomic ratio of 19.8:47.2:32.8. The surface zone showed the ratio of oxygen, nickel and titanium being 28.5:39:32.4 (red square #3, Figure 3A). For the heat-treated K3 NiTi rotary instruments, the inner zone was almost completely composed of titanium and nickel (red line #1, Figure 3B), while the intermediate zone was composed of oxygen (9.1%), nickel (65%) and titanium (25.7%) (red line #2, Figure 3B). The surface zone showed strikingly different atomic composition with the intermediate zone and inner

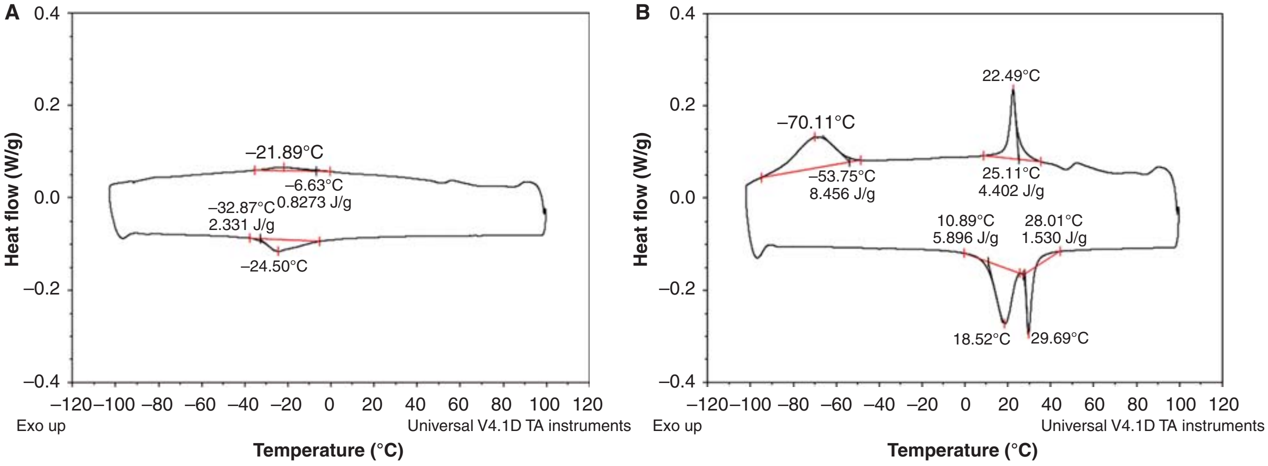

Figure 2. DSC chart of (A) as-received K3 instrument and (B) heat-treated K3 NiTi rotary instrument. Single endothermic peak with A_f of ~ 0°C was shown on heating (lower curve, A) of as-received K3 NiTi rotary instruments. On cooling (upper curve), a single Exothermic peak was observed which corresponded to the transformation from austenitic NiTi to martensitic NiTi. For heat-treated K3 files (B), two endothermic peaks were observed on heating (lower curve), which correspond to the transformation from martensitic NiTi to R-phase and subsequently R-phase to austenitic phase. The A_f of heat-treated K3 NiTi rotary instruments were ~ 45°C. Two exothermic peaks were also observed on cooling (upper curve) indicating a two-stage transformation from austenite to martensite.

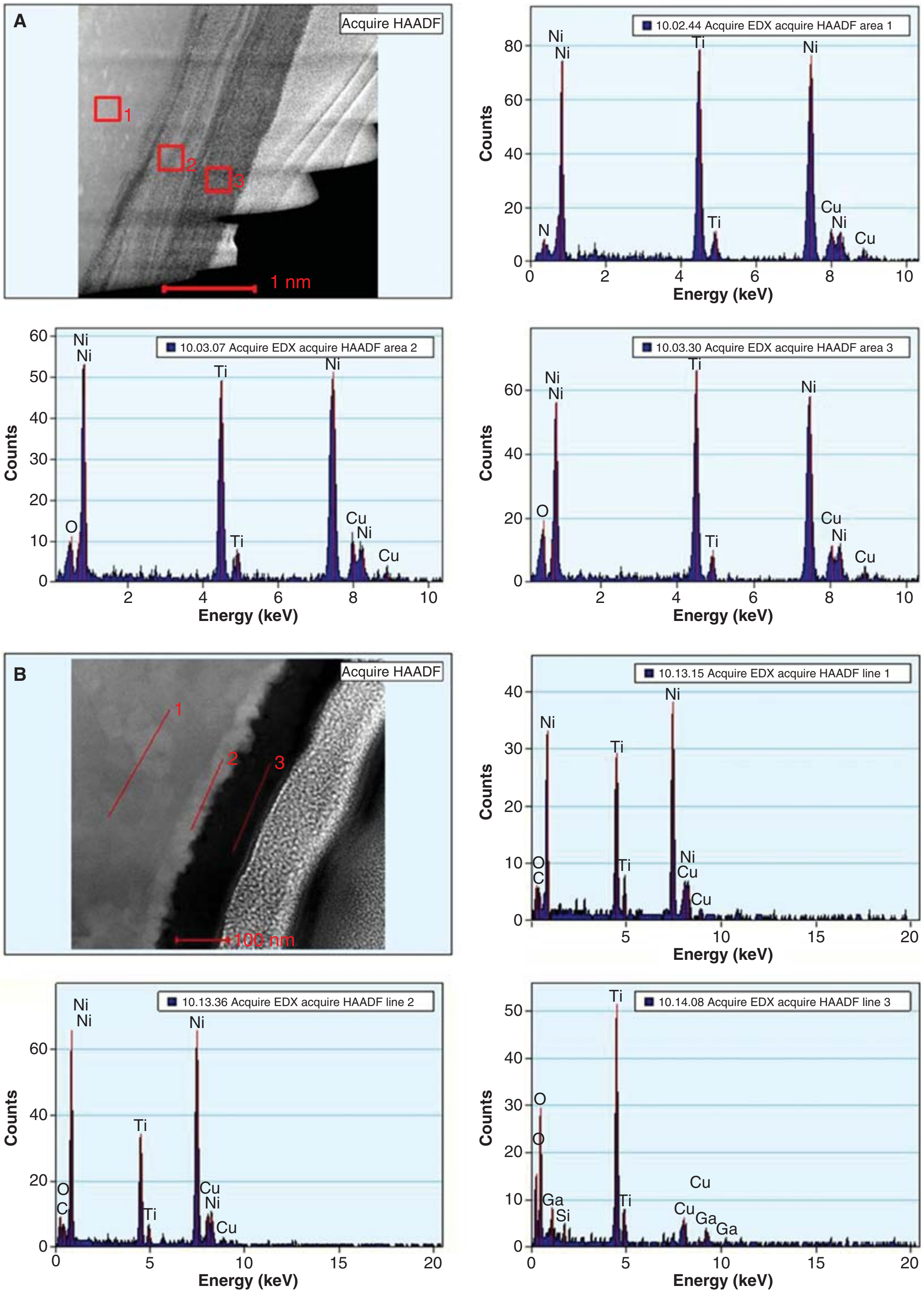

Figure 3. TEM and EDS analysis data of (A) as-received and (B) heat-treated K3 NiTi rotary instrument showing the constituting elements at different layers into the material. For as-received instrument, box 1 = inner zone; box 2 = intermediate zone; box 3 = surface zone (Figure 2A). For heat-treated specimens, line 1 = inner zone; line 2 = intermediate zone; and line 3 = surface zone (Figure 2B).

zone. The surface zone was complete composed of titanium (44%) and oxygen (55.9%).

Changes of microstructures

To identify the constituent lattice phases in the NiTi material of the instrument, selected area diffraction analysis (SADA) was performed. The results demonstrated that both as-received (Figure 4A) and heat-treated (Figure 4B) K3 NiTi rotary instruments were composed mainly of austenite, appearing as concentric circles consistent with the characteristic diffraction patterns of austenite phase. The diffraction patterns of heat-treated K3 NiTi rotary instruments

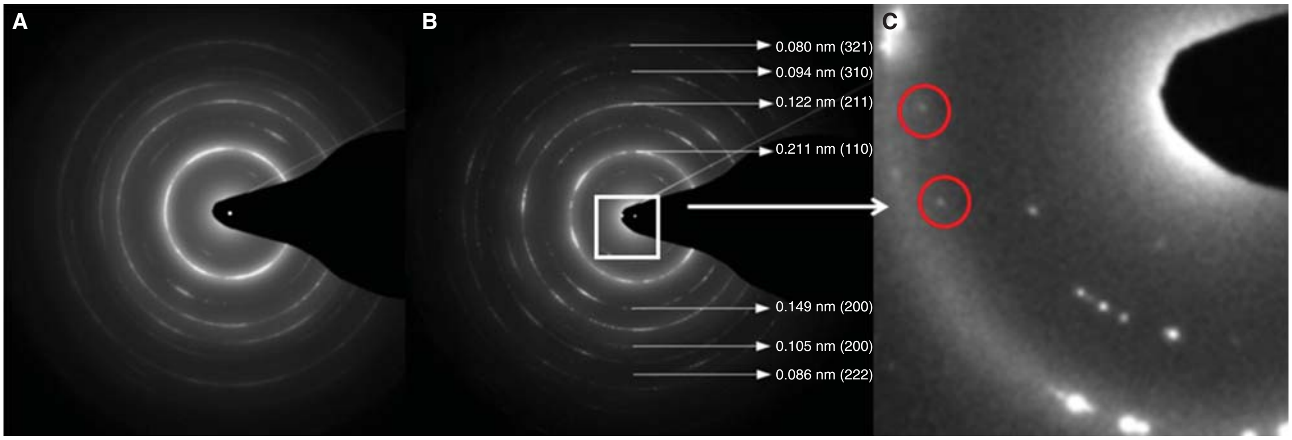

Figure 4. Selected area diffraction analysis of inner zone showing that both as-received (A) and heat-treated K3 NiTi rotary instruments (B) had a similar diffraction pattern which is characteristic of Austenite. As-received instruments showed complete concentric circles, in which the radii of the circles indicated distances between neighboring lattices. Heat-treated K3 NiTi rotary instruments showed the presence of interrupted circles, which is a result of growth in grain size. In heat-treated samples, isolated spots were observed between the concentric circles which suggested the possible existence of martensite (C).

were similar with those of as-received K3 NiTi rotary files. However, some patterns were observed in the heat-treated K3 NiTi rotary instrument which was not observed in diffraction patterns of as received K3 NiTi rotary instruments (Figure 4C).

Surface textures and grain size

The microstructure of the as-received K3 NiTi instrument (Figures 5A–C) showed that all the grains of surface zone had a size of ~ 10 nm (Figure 5B); grains in the inner zone were relatively larger (~ 100 nm; Figure 5C) than the grains in the surface zone.

The surface zone of heat-treated K3 NiTi rotary instruments showed the presence of a TiO_2 layer (Figure 5D). The grain size at the inner region of the heat-treated instruments was ~ 100 nm (Figure 5E). Existence of twin images, resembling the twinned martensite, was also observed (Figure 5F).

Discussion

The present study showed that heat treatment improved the cyclic fatigue resistance of K3 NiTi rotary instruments, compared to those of as-received K3 NiTi rotary instruments. This is in agreement with the previous study which reported the increase of

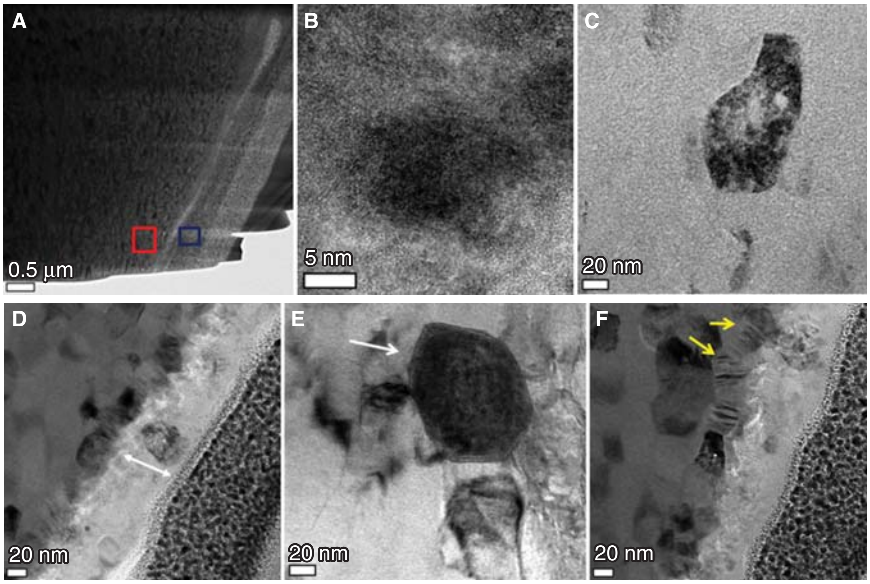

Figure 5. (A–C) TEM image of microstructures of as-received K3 NiTi rotary instruments (A). The blue area was magnified in (B), showing that a grain size near the surface is ~ 10 nm. The magnified view of red box showed that the grain size in the inner zone was ~ 100 nm (C). (D–F) Heat-treated K3 NiTi rotary instrument, showing the presence of a TiO_2 layer on the surface (D, white arrow). The grains in the inner layer had a dimension of ~ 100 nm (E, white arrow). The twin images were observed which are possibly the twin martensite (F, yellow arrows).

cyclic fatigue resistance of endodontic NiTi rotary instruments after heat treatment [12,21]. Zinelis et al. [21] have reported that heat-treated NiTi alloy showed an improved elongation limit and increased fracture strength compared with non-treated NiTi alloy and that a process temperature of 430–450°C would be optimum.

It is apparent from the results of our experiment that the as-received K3 NiTi instruments and heat-treated K3 NiTi instruments showed rather different thermal behaviors. There was a single peak with relatively small enthalpy change and A_f of ~ 0°C for as-received samples. Considering that A_f is the temperature at which austenite transformation is completed [23,24], the entire structure of as-received samples at ambient temperature would be austenitic. In contrast, the heat-treated samples showed two distinct peaks with relatively larger enthalpy change and the A_f was ~ 45°C. It is probable that heat-treated samples contain martensite or R-phase mixed with majority of austenite at body temperature. Shen et al. [26] reported that the heat-treated NiTi rotary instrument has A_f of 37°C and suggested that the material was in its martensitic phase at body temperature. The beneficial effects of martensite [26,27] or R-phase [28] on cyclic fatigue resistance of NiTi rotary instruments were reported in many previous studies. However, X-ray diffraction analysis in this study has indicated that the majority of the constituent phase of both as-received and heat-treated samples was austenitic (with a body-centered cubic structure). Therefore, it is unlikely that the increased cyclic fatigue resistance of heat-treated K3 instruments was solely attributed to the constituent phases present in the material. Rather, the increased cyclic fatigue resistance might be due to a relaxation of the residual stress introduced during manufacture by the elevated temperature. Future studies using stress–strain test would be beneficial to clarify the mechanism responsible for the increase in cyclic fatigue resistance by heat treatment.

Detailed examination of the diffraction pattern revealed that, while both as-received and heat-treated K3 instruments were essentially made up of an austenitic phase, there existed some minor differences in the SADA pattern between the two. First, the heat-treated samples showed the circles with some interruptions, while the as-received samples showed continuous concentric circles. This phenomenon appears to be related to the grain growth in heated samples. Second, there were some spots between the adjacent concentric circles in the heat-treated samples which were not seen in as-received samples. These spots might suggest the presence of another phase in a matrix of austenite which might be martensite or R-phase.

Elemental analysis of the material using EDX yielded a finding consistent with the results of SADA that the main constituent phase of both samples was austenite. TEM showed that the heat-treated samples showed relatively larger grain size than as-received instruments. Some twinned phases were observed in the heat-treated K3 NiTi instrument. It is an interesting finding that TEM/EDS analysis indicated a difference in Ni and Ti content depending on the depth from the surface where measurement was made. Areas close to the surface contained more oxygen. The heat-treated samples showed marked formation of the TiO_2 layer and Ni rich layer. The formation of TiO_2 layer on the NiTi surface is well-known and has been reported in metallurgical literature [25,29]. Although there was a report that the TiO_2 layer is important in protection of the NiTi instrument from the corroding action of sodium hypochlorite [4], the effect of TiO_2 and the Ni rich layer in NiTi rotary instruments still needs investigation.

In conclusion, the present study showed that the heat treatment of K3 NiTi rotary instruments improved its cyclic fatigue resistance and changed the thermal behavior of K3 NiTi rotary instruments that might be related to the improved fatigue resistance. The major constituting phases of both samples were austenite. However, it appears that some martensite and R-phase were present in heat-treated samples.

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Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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