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Heat Treatment and Surface Treatment of Nickel–Titanium Endodontic Instruments

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Knowledge and thorough understanding of the characteristics of endodontic nickel–titanium (NiTi) files is paramount for dentists performing root canal treatments to patients. Understanding the behavior of the NiTi files guides the clinicians in choosing the correct instruments for different clinical and anatomical situations. This review focuses on the metallurgical properties of endodontic NiTi files, with a special emphasis on recent developments and improvements in metallurgy and the effects of heat treatment and surface treatment. In this study, the impact that such developments have on the properties of endodontic NiTi files is discussed.

Keywords: heat treatment, fatigue failure, nickel–titanium instrument, surface treatment, endodontics

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INTRODUCTION

Since Walia and colleagues first introduced nickel–titanium (NiTi) instruments in the late 1980s, NiTi instruments have revolutionized the root canal instrumentation by reducing the majority of iatrogenic instrumentation issues commonly associated with stainless steel files such as zipping, ledges, transportation, and perforation (1, 2).

The first NiTi rotary instruments were marketed in the 1990s (3). Despite significant advancements in file design and manufacturing procedures for NiTi rotary instruments during the last two decades, fracture of rotary instruments induced by torsional or cyclic fatigue remains a concern for clinicians, particularly in calcified or severely curved root canals (4–6). The relative proportions and properties of the microstructural phases govern the mechanical behavior of NiTi alloy. Heat treatment (thermal processing) has been reported to influence the fatigue resistance of NiTi instruments and is one of the most common methods for adjusting NiTi alloy transition temperatures (7–10).

Nickel–titanium alloy has found a unique commercial application in the endodontic industry, because of its shape memory effect and corrosion resistance results from phase transformation. Novel NiTi instruments produced by using thermomechanical techniques, such as M-wire, R-phase, and controlled memory (CM) files, have been launched in recent years and shown to have enhanced flexibility and cyclic fatigue resistance when compared to conventional superelastic NiTi files (11–13). New NiTi instrument systems with a titanium oxide surface layer [e.g., WaveOne Gold (Dentsply Sirona, York, Pennsylvania, United States) and Reciproc Blue (VDW, Munich, Germany)] are made from NiTi alloy heat-treated in a special way.

In recent years, many new NiTi instruments have been introduced, and understanding the nature of NiTi alloy depending on their phases and their effects on instrument performance is important for clinicians for attaining favorable clinical results.

PROPERTIES OF EACH PHASE (AUSTENITIC, MARTENSITIC, AND R-PHASE)

The NiTi alloys of the endodontic instruments are made of ∼56% nickel and 44% titanium by weight, or a 1:1 atomic ratio (equiatomic) (14). Similar to other metallic systems, NiTi alloys can come in a variety of crystallographic forms. Near-equiatomic NiTi alloys have three microstructural phases (austenite, martensite, and R-phase), and their properties and their respective proportions influence the mechanical properties of the metal (15).

The austenite phase with the B2 cubic crystal structure exists at higher temperatures and is stronger (∼80–90 GPa) and stiffer than the martensite (14, 15), while the martensite phase is a low-temperature monoclinic phase (B19′) with a lower Young's modulus and yield strength (∼30–40 GPa) than the austenite phase (16, 17). This demonstrated that the martensite can be easily deformed at low stress, while the austenite has a substantially higher yield and flow stresses. The martensite phase also supports reducing the risk of file fracture under high stress conditions since it can be deformed rather than fractured. Therefore, the abundant effort has been dedicated to the introduction of martensitic alloys such as M-wire and CM wire instruments into the NiTi instruments market. Various studies investigated the performance of the instruments made from M-wire and CM NiTi and reported enhanced flexibility and fatigue resistance than those of conventional NiTi instruments (12, 13, 18).

The phase transformation from martensite to austenite and austenite to martensite can occur in one or two steps, with the two-stage transformation involving the formation of an intermediate R-phase (19). The R-phase is a "Rhombohedral phase" that differs from the cubic B2 phase in the austenite phase (20). The R-phase transformation occurs before the B2-B19′ transition and shows thermoelastic martensitic transformation features. The R-phase transformation can be induced by both temperature and stress. The recoverable strain of the R-phase/austenite transformation (∼0.5%) is smaller than that of the martensite/austenite transformation in NiTi alloy, and the temperature hysteresis is exceedingly modest (19). Furthermore, the R-phase/austenite transformation has remarkable cyclic stability and Young's modulus of R-phase is lower than that of the austenite (21).

THE PHASE TRANSFORMATION OF NiTi ALLOY

Conventional superelastic NiTi alloys present in the austenite form at room temperature. As austenite cools down, it begins to transform into martensite at the martensite transformation start temperature (Ms) and completes the transition at the martensite transformation finish temperature (Mf). On the other hand, when martensitic NiTi is heated past the austenite transformation start temperature (As), the crystal structure of the NiTi begins to transition to austenite, and once heated past the higher austenite finish (Af) temperature, the NiTi crystal structure becomes entirely austenite (**Figure 1**).

The phase transformation from austenite to martensite can also be caused by stress or external force, which can accommodate greater stress without increasing strain (14, 22). Compared to stainless steel, the superelasticity of NiTi allows the total recovery of the deformations up to 8% of the deformation (14). As a result, a conventional NiTi instrument, in an austenitic state at body temperature, exhibits transformational elasticity or the capacity to return to its original shape after being distorted. When external stress such as torsional stress or file friction against canal walls is applied, the stress-induced martensitic transformation occurs, resulting in more resilient materials with a higher ultimate tensile strength (23). Because the stress-induced martensitic state is not stable at room temperature, once the stress is relieved, the deformed NiTi alloy immediately reverts to the austenite phase. The NiTi files can shape the root canal with a constant cutting force in this manner, even in a curved root canal. When the NiTi alloy is deformed in the martensite state by an external force, it can be also fully recovered when heated. Deformation via martensite reorientation can be observed at temperatures below As, the starting temperature which is important for the reverse transformation of martensite upon heating and is completed at Af (19).

SUPERELASTICITY AND SHAPE MEMORY

Nickel–titanium alloys show unique superelasticity and shape memory properties (14, 24). When the ambient temperature is higher than the Af temperature of the NiTi alloy, superelasticity is interrelated to the occurrence of a phase transition of the alloy when stress is applied above a critical level. When the stress is relieved, the stress-induced martensitic change reverses spontaneously and the material returns to its previous shape and size (24). In other words, when the endodontic instrument is removed from the root canal, it reverts to its original shape (25). The enhanced flexibility of NiTi instruments over stainless steel instruments is due to this reversible thermoelastic martensitic transition, which makes the instrumentation of curved root canals easier and safer (14). Superelasticity occurs when austenite and martensite undergo a reversible phase change. As a result, transformation temperatures have a significant influence on the mechanical characteristics and behavior of NiTi, which can be influenced by minor compositional changes, impurities, and heat treatments during the manufacturing process (26).

HEAT TREATMENT OF NiTi ALLOYS

The goal of heat treatments is to change the transition temperatures of NiTi alloys and, as a result, modify fatigue

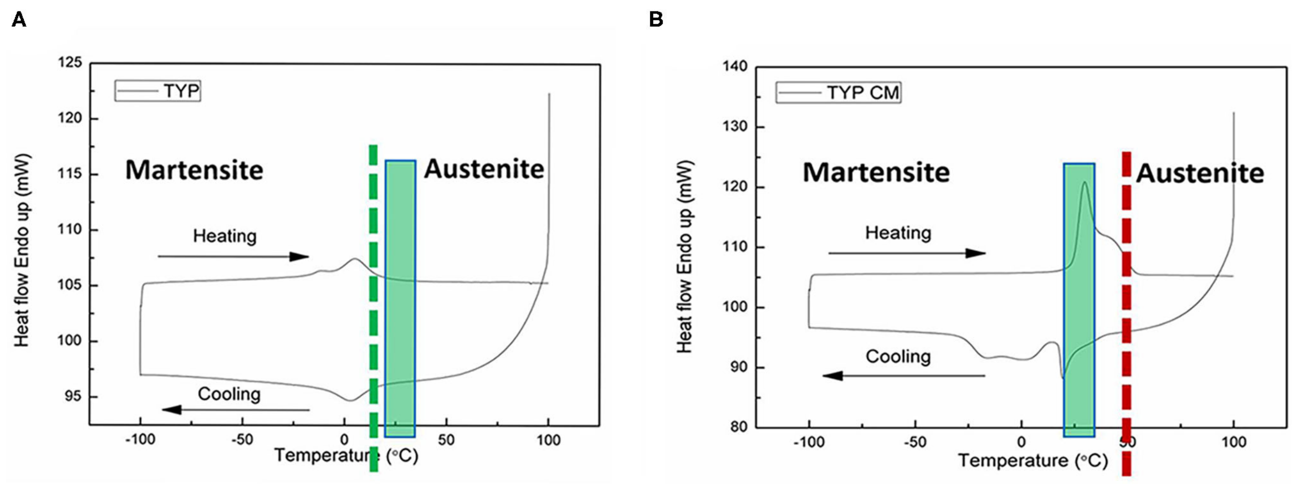

FIGURE 1 | Differential scanning calorimetry curves of nickel–titanium (NiTi) instruments. The austenite-finish (A_f) temperature shown with blue dotted line is 17°C for a "conventional" superelastic NiTi file **(A)** and 55°C for heat-treated NiTi file shown with a red dotted line **(B)**. The blue bar represents the temperature range between room temperature and body temperature.

resistance. Superelastic conventional NiTi instruments existed in the austenite phase at room and body temperatures, which limited their usage in severely curved canals due to the stiffness of the instrument and low fatigue resistance (7, 27). The heat treatment process releases the internal strain of NiTi alloy and increases the phase transformation temperature of NiTi, resulting in more martensite phase at clinically relevant temperatures (7, 10), which makes heat-treated NiTi instruments higher flexibility and fatigue resistance than those of conventional NiTi instruments (**Figure 2**).

In early 2000, a new method for optimizing the structure of NiTi wire blanks for rotary instruments has been developed. Several proprietary thermomechanical processing techniques have been established with the goal of creating superelastic NiTi wire blanks that contain the significantly stable martensite phase in clinical conditions. M-wire (Dentsply Tulsa Dental Specialties) was introduced in 2007. It was developed by applying a series of heat treatments processes and contains three phases: martensite, R-phase, and austenite (11). M-wire instruments include Dentsply's ProFile GT Series X, ProFile Vortex, ProTaper Next, and WaveOne. In 2008, a new manufacturing process was developed by SybronEndo: Twisted Files (TF). TF is manufactured by twisting the NiTi rod, while most NiTi files are manufactured by the grinding method. The manufacturer claims that TF instruments were created by thermally transforming a raw NiTi wire in the austenite phase into the R-phase. R-phase occurs within a very narrow temperature range on the heating or cooling curve between martensitic and austenitic forms and made it possible to twist the NiTi rod. Previous studies reported that TF has greater cyclic fatigue resistance than files that have been manufactured by grinding, while the torsional resistance of R-phase files was significantly lower than that of ground files (28–30).

In 2010, CM wire (DS Dental, Johnson City, TN, United States) was introduced as a new NiTi alloy with high flexible properties. CM NiTi files are made by a specific thermomechanical technique that controls the memory feature of NiTi alloy, making them exceptionally flexible but lacking the shape memory which is seen in other superelastic NiTi files. In other words, CM NiTi files do not rebound after unloading, and their original shape is restored following the application of heat. Thermally treated CM alloys would be primarily or entirely in the martensite phase at body temperature because the Af temperature of CM wire is ∼55 and 50°C (7–9, 102). CM NiTi files include HyFlex CM and EDM (Coltène/Whaledent, Altstätten, Switzerland), Typhoon Infinite Flex NiTi Files (Clinician's Choice Dental Products, New Milford, CT, United States), and VTaper 2H (SS White, Lakewood, NJ, United States).

Thermal treatments have been reported to influence the mechanical properties and transformation features of NiTi alloys based on their thermomechanical history (31). Heat treatment would be applied prior to machining the instrument to reduce the work hardening of the alloy (32, 33). Recently, the application of this heating process also has been applied after the machining of the files, with the aim of transforming the alloy into a slightly different crystalline phase structure with enhanced mechanical properties (improved flexibility with superior mechanical resistance) (33). Post-machining heat treatment is applied to K3XF (SybronEndo, Orange, CA, United States) instrument. K3XF showed similar torsional properties, but greater flexibility and resistance to cyclic fatigue than those of the original K3 instrument (33, 34).

In comparison to conventional superelastic NiTi rotary instruments, heat-treated NiTi instruments have greater flexibility and cyclic fatigue resistance (7). It can be assumed that the cutting efficiency of the comparatively soft and flexible

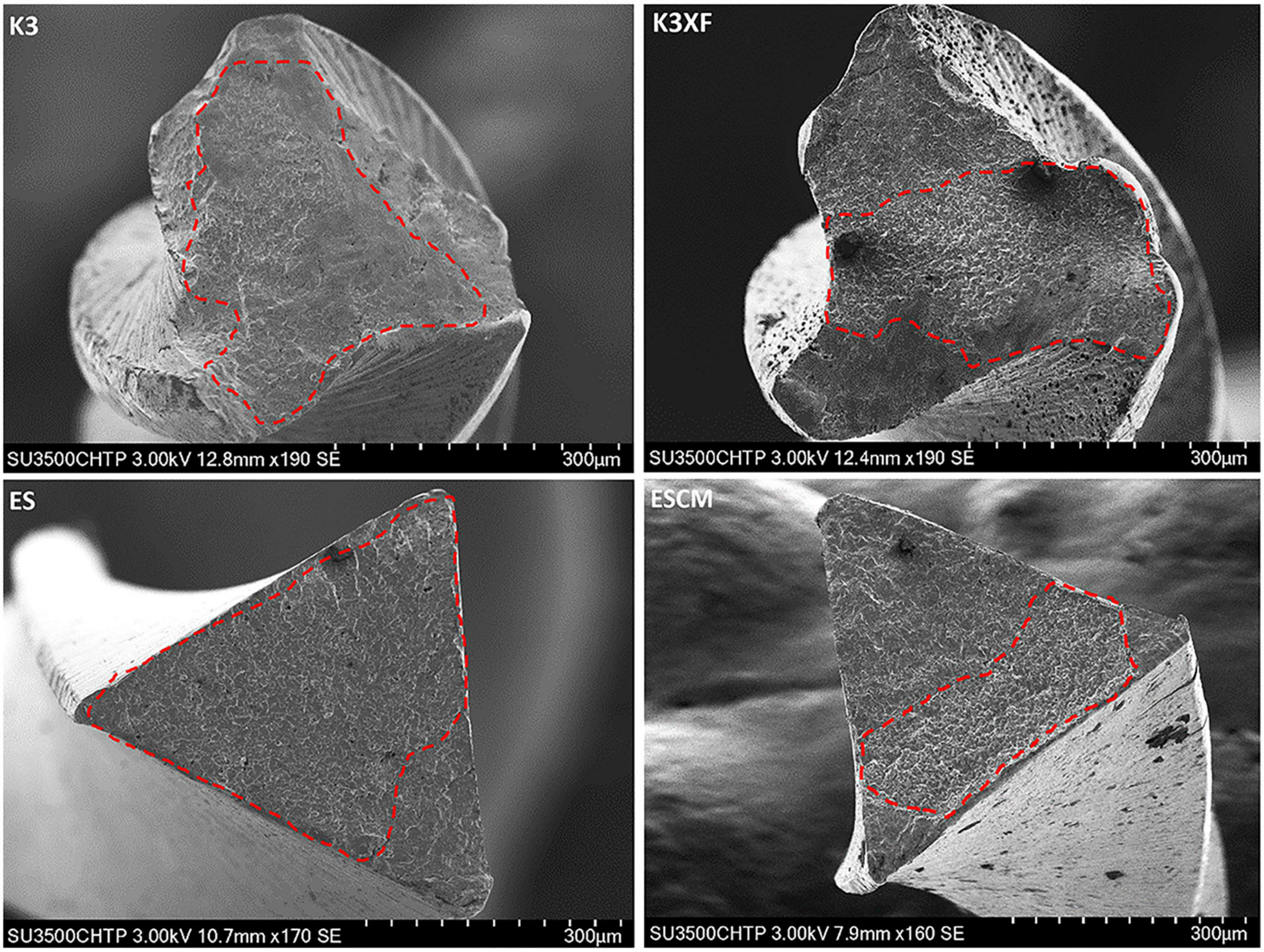

FIGURE 2 | The photomicrograph of the fracture surface of NiTi instruments with the region of crack propagation and dimple area outlined (dotted line).

NiTi instruments is lower than that of the relatively stiff NiTi instruments. However, two investigations (35, 36) indicated that a heat-treated NiTi instrument (HyFlex CM) showed the most efficient cutting instrument in lateral action against dentine and acrylic resin when compared to other coronal flaring instruments such as BioRace (FKG Dentsaire SA, La Chaux-de-Fonds, Switzerland), ProFile (Dentsply Maillefer, Ballaigues, Switzerland), and ProTaper (Dentsply Maillefer).

SURFACE TREATMENT OF NiTi ALLOYS

Surface treatment of NiTi instruments reduces inherent defects, increases surface hardness and flexibility, and improves fatigue resistance and cutting efficiency (37–39). Microcracks are frequently formed on the surface of the instrument, indicating the very first stage of the fatigue phenomenon (40). Thus, a treatment that improves surface smoothness is expected to inhibit crack initiation and increase fatigue resistance. Electropolishing refers to any electrochemical procedure that aims to reduce the surface irregularities of material and achieve a high gloss finishing. It is carried out by immersing the part in a specially formulated, usually acidic, electrolyte solution and passing a direct electric current to facilitate a selective dissolution of the material (39). RaCe (FKG Dentaire) and EndoSequence (Brasseler, Savannah, GA, United States) NiTi file systems have undergone the electropolishing process. Previous studies (41, 42) have demonstrated that electropolishing improves the fatigue resistance of NiTi instruments, while some other studies have shown that the benefits of electropolishing may vary depending on the instrument type, design, and cross-sectional area (43, 44).

The surface hardness and wear resistance of heat-treated NiTi instruments have been reported to be improved using surface engineering techniques. Physical vapor deposition describes a variety of vacuum deposition methods that can be used to produce thin films and coatings. Several manufacturers have devised thermomechanical processing sequences to generate a titanium oxide surface layer for the NiTi instrument. Gao et al. found that the comparatively hard titanium oxide surface layer of the Vortex Blue (Dentsply Tulsa Dental, Tulsa, OK, United States) instrument may compensate for the loss of hardness when compared to ProFile Vortex M-wire while enhancing cutting efficiency and wear resistance (45). HyFlex electrical discharge machining (EDM) is manufactured via EDM, a non-contact thermal erosion process that partially melts and evaporates the wire by high-frequency spark discharges and shows higher resistance to cyclic fatigue than HyFlex CM (46).

FUTURE PROSPECTS

Endodontic hand- and engine-driven NiTi files have been available for clinicians already for almost 30 years. Continuous development has taken place since the introduction of the first NiTi files. It is highly likely that this path of incremental improvements will continue in the foreseeable future. However, there is currently no specification or international standard for assessing the fracture resistance of endodontic rotary instruments. Despite the fact that the cyclic fatigue and torsional test do not accurately represent clinical settings, it is required for evaluating the mechanical properties of endodontic instruments. By modifying the microstructure of the NiTi alloy through heat treatment, the mechanical properties of the alloy can be enhanced. Because the heat-treated files have a higher resistance to cyclic fatigue as well as an increase in ductility, the incidence of file fracture during clinical use might be reduced (46, 47). It is assumed that the higher ductility assessed by angular distortion gives the heat-treated instrument a higher "safety factor," because files with more observable distortion of the cutting spirals are more likely to be discarded before breakage (48). However, a decrease in cutting efficiency has been reported in the heat-treated instruments (49). One of the potentially interesting recent observations is the effect of cold on the fatigue resistance of NiTi files (50). Whether this result can be transferred to the clinical situation of the root canal, remains to be seen. As rotary NiTi files fracture mainly due to limited fatigue resistance, it can be assumed that success in improving this characteristic of the NiTi files will be a key factor in future improvements of NiTi files in the instrumentation of the most difficult anatomies.

CONCLUSIONS

The changes in transformation behavior as a result of heat treatment have been found to affect the mechanical characteristics, enhancing clinical performance compared with files of similar design and size made from conventional NiTi alloy. Heat-treated and CM NiTi instruments are frequently employed by clinicians for endodontic treatment nowadays. Although the details of the thermomechanical treatment history of the new NiTi wires are unknown until now, it appears that thermomechanical processing is a very promising method for improving the efficiency and safety of contemporary endodontic instruments. However, it is important to remember that all instruments have strengths and weaknesses and that properties are determined by a variety of factors such as alloy type and degree of taper and cross-sectional design.

AUTHOR CONTRIBUTIONS

SK contributed to the drafting and the critical revision of the manuscript. MH contributed to the conception, designing, and the critical revision of the manuscript. HCK contributed to the critical revision of the manuscript. ZW and HL contributed to the drawing of the figures and the critical revision of the manuscript. YS contributed to the conception, designing, drafting, and drawing of the figures as well as the critical revision of the manuscript. All authors contributed to the article and approved the submitted version.

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