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Endodontic instrument, metallurgy, NiTi alloy, thermomechanically heat treatment

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Crystallographic Metallurgy of Nickel-Titanium Instruments in Endodontics: A Comprehensive Review

Abstract:

A solid grasp of the mechanical and physical properties of nickel titanium (NiTi) endodontic files, as well as their link to metallurgical characteristics is required for clinicians to appreciate the role and proper use of NiTi instruments during root canal treatment. Various thermo mechanical treatment techniques have been created to increase the clinical performance and improve the fracture resistance of NiTi instruments. Knowledge will also assist clinicians in selecting the most appropriate instruments for root canal treatment. During heat treatment, NiTi alloy undergoes phase change to increase mechanical characteristics. This narrative analysis evaluate how various thermo mechanical heat treatment technique affect the metallurgical properties of NiTi alloys, as well as discussing newly introduced thermomechanical process altered NiTi endodontic instruments.

Introduction:

Since the time of Walia et al., nickel titanium (NiTi) alloys have been utilized in the last 3 decades in endodontics and they have brought about a major breakthrough in root canal treatment (16). NiTi endodontic instruments were more flexible than stainless steel (SS) instruments due to enhanced resistance against torsional fracture (46). These enhanced qualities resulted in a significant improvement in engine or machine driven endodontic instruments (2).

In contrast to SS instruments, the use of NiTi instruments in engine or machine driven endodontic handpieces reduces the likelihood of procedural errors (3). However, despite the improved metallurgical properties, NiTi instruments are still vulnerable to be brittle during the biomechanical preparation of root canals (3). During mechanical instrumentation within the root canal, two forms of fracture of NiTi devices can occur: flexural fracture and torsional fracture (4). NiTi instruments may suffer a flexural fracture during biomechanical instrumentation of curved root canals due to an increase in their cyclic fatigue (4). Torsional fractures develop as a result of constant phase transformation inside the material produced by the repeated loading and unloading of the instrument during function (4,5). Applying stress to the instrument causes microstructural changes, which produce phase transformation. Several thermomechanical modifications of the alloy can have an impact on these phenomena (6).

Numerous patented thermal processing techniques for NiTi alloys have been designed to enhance their mechanical properties (9). In addition to thermal and mechanical treatment techniques, manufacturers have also implemented some machining procedures such as the twisted method and the electrical discharge machining method and chemoelectrical surface finishing procedures (10).

NiTi alloys used for endodontic devices are classified into two types by major phases: those with a high concentration of austenite phase (traditional / conventional NiTi instruments, R-phase, M-wire) and the others with a high concentration of the martensite phase (Gold and Blue heat treated NiTi, controlled memory wire) (9).

When the proprietary thermo mechanically processed NiTi alloys were compared with the conventional NiTi, the thermo mechanically treated NiTi alloys demonstrated increased flexibility, superior cycle fatigue resistance, and a larger distortion angle before torsional failure (7). These improved properties might be ascribed to a modified phase including different proportions of martensitic alloy as well as R-phase alloy (11). Endodontic instruments composed of austenitic alloys have superelastic (SE) capabilities

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due to transformation from the stress induced martensite and they will revert to their native state after deformation (2). On the other hand, due to the reorientation of the martensite properties, the martensitic devices are easily distorted, and the form memory effect can be seen once heated (12). When compared to austenitic alloy, martensitic alloy produces a range of more flexible instruments with enhanced cycle fatigue resistance (5). The newly developed thermomechanically treated NiTi instruments have had an evolving and progressing impact on mechanical properties due to enhanced fracture resistance and overall improvement in rotary endodontic instruments' clinical performance (5,13).

This review summarises the history of NiTi alloy used for endodontic files and the developmental advancement of the instruments with clinical implications.

History of nickel titanium (NiTi) alloy

NiTi alloy was first developed in 1963 at the Naval Ordnance Laboratory for the United States space program and dubbed as "Nitinol" at the time (10). Andreasen and Hilleman reported its first usage in the production of orthodontic wires in 1971. Walia, Brantley, and Gerstein then introduced the first hand NiTi file in 1988, which was made by notching orthodontic wire (14). The shape memory property and super elasticity of NiTi instruments are due to its micro structural phase transformation (15).

NiTi alloy made up nickel (56%) and titanium (44%).⁽¹⁵⁾ It exists in two major phases that are determined by their crystal structure (5,10):

- The Austenitic phase is a parent phase that possesses cubic B2 crystal structure.
- The monoclinic B19 crystal structure is made up of the Martensitic phase.

There is also a transitional phase, known as the Rphase (9). The nature and relative proportions of the components affect the mechanical characteristics of the metal (16). When compared to the martensitic alloy, Austenitic alloy is harder and has a better superelastic property (17). Based on this property, martensitic alloy is flexible, adaptable and has a shape memory effect. It also more resistance to cyclic fatigue than austenitic alloys (Fig. 1) (9).

The alloy will be in its austenitic state, if the transition temperature is higher than the austenitic finish (Af) temperature (2). If the temperature drops below the martensitic finish (Mf) temperature while cooling, the alloy is in the martensitic phase or daughter phase (10). The thermo elastic behavior of a martensitic alloy can develop either through the administration of stresses, resulting in strains known as stress induced martensite (SIM), or by a heat change known as thermally induced martensite (TIM) (Fig. 2) (9). Detwinning allows martensite's original form to be readily bent to a single orientation structure known as detwinned martensite (15). The austenite phase is elastic in comparison to the martensitic phase

(10). If the temperature produced is above the temperature transformation range, heating the alloy can reverse the deformation (i.e. the reverse temperature transformation range) (9,10).

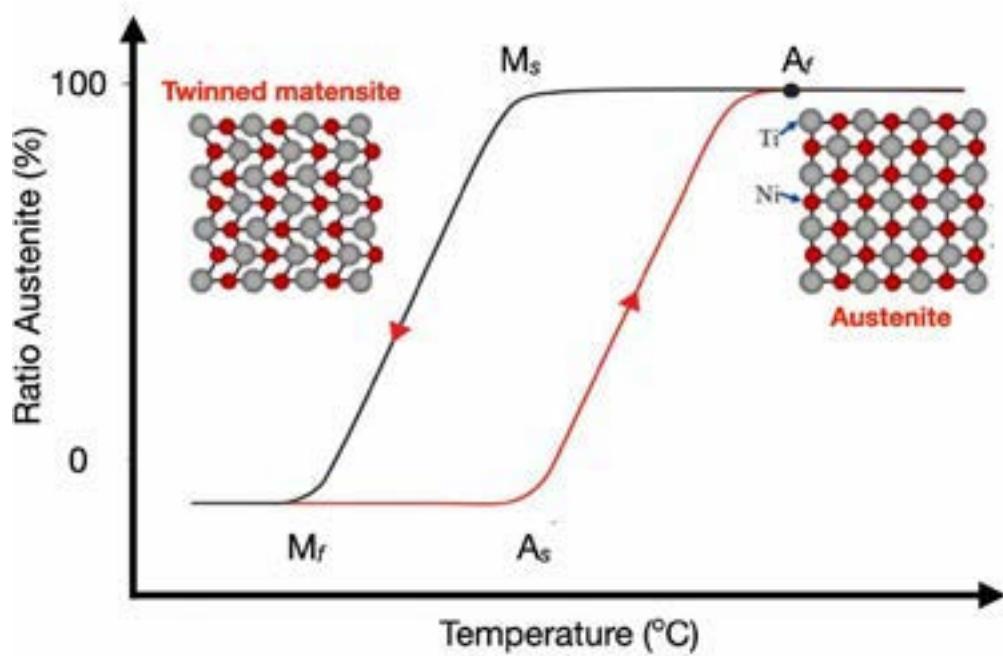


Figure 1. The temperature dependent transformation from the austenitic phase to the martensitic phase for a nickel titanium alloy. The Xaxis represents temperature in Celsius (°C), and the Y- axis represents the ratio of austenite (%). (Ms: Martensite start temperature, Mf: Martensite finish temperature, As: Austenite start temperature, Af: Austenite finish temperature)

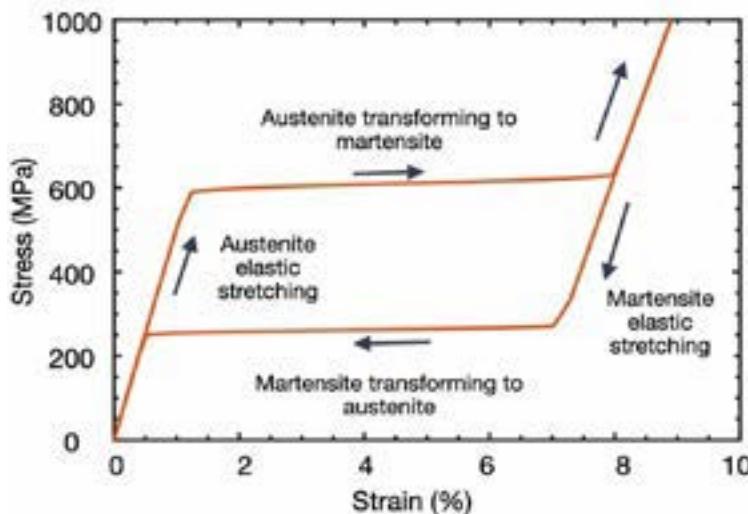


Figure 2. NiTi alloy phase transformation process

Fracture resistance of NiTi alloy

The possibility of NiTi instruments fracture is a significant risk in clinical endodontic treatment.(4) Fractures are frequently caused by inappropriate or excessive use of the device. NiTi instruments can fracture in two ways: (i) torsional fracture and (ii) flexural fracture (4,15,18).

Torsional fracture is typically caused by the application of a high tensional force to the instrument (5). Constant loading and unloading of an instrument causes significant pressure tension, which may surpass the alloy's 8% limit. This will result in plastic deformation that is irreversible, resulting in fracture (6).

Flexural fracture is caused by cyclic fatigue of the instruments (19). Cyclic fatigue occurs when the instrument is exposed to several tension and compression cycles (14). After putting a load to the equipment, scanning electron microscopy (SEM) revealed irreversible microcracks on the alloy's crystallographic structure (20), followed by crack propagation resulting in fracture (5,20).

Phase transition behaviour has a significant impact on the mechanical characteristics of NiTi instruments. Several elements, including manufacturing method, heat treatment, and chemical composition, have a significant impact on it (21). By performing phase transformation during the heat treatment process, the instrument's flexibility may be increased, which improves the metallurgical properties of NiTi files by enhancing their resistance to cyclic fatigue (14).

Effects of heat treatment on phase transformation properties and crystalline microstructure

Temperature affects how NiTi alloys reacts. NiTi will be in an austenitic condition characterized as a centered cubic lattice after higher temperature processes (9,18). At lower temperatures, NiTi has a martensitic crystalline structure with a monoclinic deformed structure (7). This deformed structure allows for higher angles of deformation than the austenitic NiTi state (3). Stress and heat cause phase shifts. Complex procedures like as Ms, Mf, As, Af, Rs and Rf have been used by manufacturers to adjust the alloy transformation temperatures, thereby improving its mechanical performance (17).

One of the primary advantages of using NiTi is the instruments' tremendous flexibility (22). The instruments have SE properties, which cause reversible deformation when stress is applied (23). To demonstrate the superelastic characteristic, the alloy should be employed in its parent austenitic phase

(24). At normal room temperatures, the austenitic phase is present in conventional NiTi alloys (7). When austenitic NiTi is activated, it undergoes linear stress/strain elastic deformation (6). As the stressinduced deformation develops, the SE deformation occurs, while the strain remains constant (25). This fundamental advantage of the martensitic transition observed at the crystallographic level is this superelasticity (26).

Developmental generations of endodontic instruments

The first generation (Superelastic files)

McSpadden created the first rotary system with 2% or 0.02 taper size of NiTi instrument which was commercially available in 1992 (14). Despite the fact that new instruments started to alter dentists' perception on root canal instrumentation, issues with file breaking persisted (5). Johnson first launched the ProFile 0.04 tapered series of files in 1994 (14). ProFile 0.06 tapered files and the Orifice Shapers were created shortly after. Milling three regularly spaced Ushaped grooves throughout the entire shaft of a tapered NiTi wire produced the crosssectional forms (27). The radial land area was defined as an unground area close to every groove to achieve this design (22). The flat section was designed to keep the file from catching or locking inside the root canal. A passive planing action created the cutting effect (28). Shortly after, various rotary files systems were developed, each with its own set of alleged advantages – these file systems included Greater Taper (GT) files, Quantec file system, and LightSpeed system (29).

The earliest generation of files alloys were austenitic, with Austenite finishing (Af) temperatures lower than body temperature and SE characteristics (30). The Af for most SE NiTi files was at or under ambient temperature (1631°C), and the devices were in the austenitic SE state, according to metallurgy of SE files investigations utilizing standard differential scanning calorimetric (DSC) research (14). ProFile

(Dentsply Tulsa Dental, Tulsa, OK) and LightSpeed (Lightspeed Inc, San Antonio, TX, USA) were reported to have an Af of approximately 25°C (6). EndoSequence (Brasseler, Savannah, GA, USA) and Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT) files had Af temperature of 31.13°C and 16.22°C, respectively (9). According to another study, the Af for ProFile GT was 21°C, 17°C for ProTaper Universal (Dentsply Tulsa Dental, Tulsa, OK, USA) and 3.88 ± 3.21°C for K3 (SybronEndo, Orange, CA, USA) (31). All earliest NiTi rotary files included passive cutting capabilities, such as peripheral surfaces and a fixed tapers system. By using these file systems, to satisfy the objectives of root canal preparation, multinode number of files were necessary (13).

The second generation

In the end of the 1990's, the commercial availability of the second generation of NiTi rotary files was announced (24). The most significant distinctions between this generation of files were the presence of active trailing edges without radial lands as well as the use of fewer instruments for canal preparation

(5). The angle formed by the instrument's longitudinal axis and the cutting blade was less than in first-generation files, lowering the possibility of a "screwing in" effect throughout operation (2,32). The ProTaper (Dentsply Tulsa) rotary files of the second generation included several and varied tapers (progressive changing taper) along the length of each file, in contrast to all other passive or active NiTi cutting devices (33,34).

Several patented thermomechanical processes for producing SE wire blanks were developed, and they were found to be in the stable martensitic phase under normal clinical settings (5). During manufacture, thermal processing of the alloy improved crystal structures and changed the relative percentages of existing alloy phases (31). Heat treatment often results in finely distributed NiTi particles in the matrix as well as an increase in the alloy's Af, leading in different crystallographic percentages of martensite, Rphase, and/or austenite close to body temperature (2,26). Three unique wires will be generated based on the thermodynamic processing of the wires prior to or during manufacture (14).

The third generation

NiTi metallurgical advancements resulted in the third generation of NiTi files (5,15,18,31). Heat treatment (or the heat processing techniques) is among the most popular and essential methods for controlling both the transition temperatures and the fatigue tolerance of NiTi alloys (28). Since 2007, there has been a significant advancement in the thermomechanical processing and manufacturing for optimizing the microstructure of NiTi alloys (31).

To overcome faults introduced by cutting the files to modify the structure of the crystalline phase, a novel method of heating was introduced following the machining process (35). It has been found that the martensitic transformation of NiTi alloys happens in 2 stages instead of one during heat cycle. In nickelrich NiTi alloys, the first stage transition (AM) happens, while the second stage transition (ARM) happens after the additional heat treatment (36). The thermal treatment causes finely dispersed Ti3Ni4 crystallized to develop in the austenitic matrix (2). Because of the presence of the tiny Ti3Ni4 pieces, the Rphase with a propensity for martensite is formed (37). Because the alloy requires extra cooling to generate martensite, the marten-

sitic deformation happens in two stages (ARM) (2,16,37).

The fourth generation

The large proportion of widely available NiTi files used for root canal treatment are mechanically operated in endodontic handpieces by a continuous rotating movement (23). However, certain files are used in a reciprocating fashion, with repetitive backandforth action. As early as 1958, SS versions of similar files were produced (7). At first, all rotational motors and handpieces moved in equal 90° clockwise (CW) and 90° counterclockwise (CCW) arcs. Reciprocating systems have evolved over time to use smaller but equal, arcs of CW/CCW rotation. Reciprocating systems with equal CW/CCW arcs of 30° include the M4 (SybronEndo), EndoEzeAET (Ultradent), and EndoExpress (Essential Dental Systems) files (38).

Contemporary WaveOne and Reciproc (VDW) were introduced as singlefile instrumentation systems (35). They move in asymmetrical reciprocal arcs (14). The CCW engaging arc is smaller than the file's elastic limit and five times the CW detaching arc (28). After 3 CCW/CW cutting cycles, the file will have completed a full rotation of 360° revolution, or a full circle. This reciprocating motion enables a file to function better and proceed more easily to the apical end of the canal while cutting and auguring particles out of the canal (31,39).

The fifth generation

The fifth generation of NiTi files uses a snake movement like a wave motion along the active cutting portion of the files (5). They were manufactured with an offset mass center (Centreoff design) and/or the rotation center which results in a wavelike motion during rotation.(3) The goal of this offset is to reduce the conflict that exists between the file and the dentin. Additionally, it enhances debris removal from the root canal and promotes flexibility along the active area of the file. Variations of this concept can be found in the file systems such as RevoS (Medidenta), One Shape (MicroMega, Besançon, France), and ProTaper Next (Dentsply Sirona) (40).

ProTaper Next system provides 5 files available in varying sizes: X1 (17/0.04v), X2 (25/0.06v), X3 (30/0.07v), X4 (40/0.06v), and X5 (50/0.06v) (17). These tapers represent the taper of each file's tip area and they vary throughout the active section of the file (41). Both the PTN X1 and X2 files have a growing and decreasing percentage taper, however PTN X3, X4, and X5 files feature a fixed D1D3 taper, then a falling proportion taper over their remaining active sections (24). PTN files incorporate three important design elements – many tapers on a single file, Mwire technology, and the offset design (7). Lowering the instrument's engagement with the dentin limits both the unwanted taperlock (also known as the screwin effect) and the torque on the file. The offset design may help reduce the chance of debris laterally compacting into the canal wall, which might contribute to canal obstruction (22).

The RevoS NiTi system includes 3 shaping device: the forming and cleansing device (SC) number 1 (SC1)(#25/0.06), the SC2 (#25/0.04), and the universal shaper (#25/0.06).(6) The irregular crosssection design allows for snakelike movement through the canal which is believed to alleviate torsional stress on the file (31). According to the manufacturer, this sequence slices dentin and eliminates debris, followed by a cleaning cycle that facilitate coronal

clearance of the dentin debris generated by the cutting.(40) The only singlefile NiTi instrument recommended for use with continuous rotation is the One Shape file (6). It has a varying crosssection along the cutting tool, allowing for optimal cutting process in three areas of the canal wall. The very first zone has a varying 3 cuttingedge design, the second has a crosssection prior to the transition that gradually shifts from 3 to 2 cutting edges, and the third zone has 2 cutting edges (used in the coronal portion of the canal) (14).

Variation of NiTi alloy by heat treatment Mwire

The substance used for thermal treatment of Mwire contains $55.8 \pm 1.5\%$ wt nickel (Ni), $44.2 \pm 1.5\%$ wt titanium (Ti), and trace elements less than 1%wt.(16) Mwire's austenite finish (Af) temperature ranges between 43° and 50°C , showing that it is not totally built of austenite when used in clinical tooth treatment (15). At body temperature, Mwire has an austenite phase with a trace of martensite and Rphase (2). Mwire is more flexible than the traditionally processed SE NiTi wire. In addition, martensite and Rphase have less moduli of elasticity than austenite (40). Therefore, the presence of these two phases can be linked to the Mwire's improved flexibility (42). According to an analysis of the stressstrain curve of Mwire, less pressure is needed to trigger martensite transition in Mwire than in ordinary NiTi (17). Mwire has been found to be significantly less susceptible to cyclic fatigue than typical NiTi wire while

maintaining comparable torsional properties (14,42).

Rphase

SybronEndo (Orange, CA, USA) designed Twisted File (TF), a novel rotating NiTi technology, shortly after the introduction of Mwire in 2008 (9). The production process includes three steps: Rphase thermal treatment, alloy wire twisting and a specific surface conditioning. A raw austenitic NiTi wire is turned into Rphase for the twisting procedure using a proprietary thermal processes (28). The Rphase has a lower shear modulus and a transformation strain which is less than a tenth of martensite transition (3). Twisting process requires less tension to generate plastic deformation in the Rphase. After bending, TF is changed back to austenite by further heat processes in order to retain its new form. The austenite final temperature of Rphase tools ranges between 18° and 25°C , implying that when used clinically in the mouth, these instruments primarily contain SE austenite (24).

Rphase instruments have already been revealed to have superior cyclic fatigue resistance and flexibility when in comparison to standard NiTi instruments manufactured without heat treatment (31) and comparable cyclic fatigue resistance to Mwire instruments (20). Rphase instruments have a larger angle of bending at fracture but a lower maximum torque when contrasted to MWire and conventional NITi devices (Fig. 3)(11). They have also been shown to provide more central root canal preparations with less mobility than standard NiTi rotating processes (11).

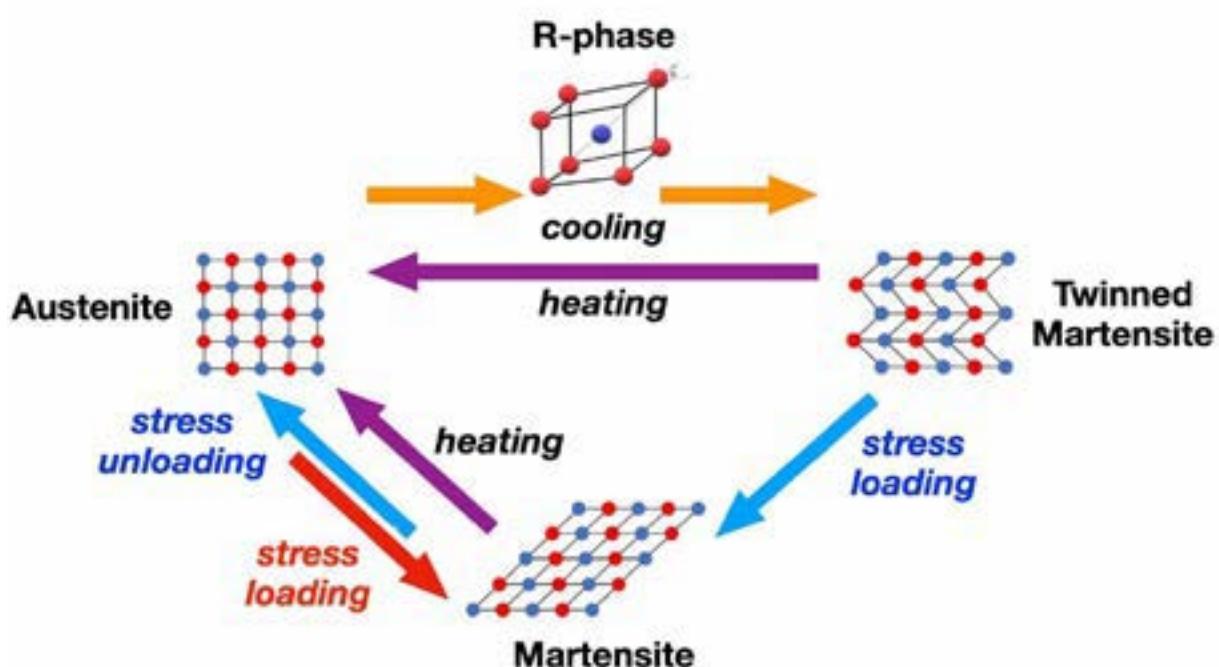


Figure 3. Schematic illustration of phases transitions of the NiTi lattice structure by stress and temperature; Cubic shaped of B2 austenitic phase, twinned and detwinned martensite, and distorted rhombohedral Rphase

Martensitic alloy

Cooling under a crucial transformation temperature range, causes electron bonding to modify the modulus of elasticity, yield strength and electric resistance of NiTi alloy (Fig. 3) (18, 43). Lowering the temperature leads to an alteration in the crystal structure referred as the martensitic transformation (44) which gives the alloy with its shape memory effect (33).

The degree of alteration is determined by the starting (Ms) and the finishing (Mf) temperatures (26). The shear kind of process causes the alloy undergo transformation, resulting in changing metallurgical properties and the production of a new stage known as the martensitic or daughter step. This results in twinned martensite, which has the structure of a tightly compacted hexagonal structure (18). This martensite structure is easily distorted

to a singular orientation by a process known as detwinning when subjected to a "flipping over" form of shear (9). Another feature of the device in this transformed martensitic phase is that when heated above the austenite final (Af) temperature (e.g., autoclaving), the alloy returns to its native form by reverting to the basic austenitic condition (45). Compared to the austenite phase, the alloy is very ductile and soft in the martensitic phase (24).

Controlled memory (CM) wire

Controlled memory (CM) wire was announced in 2010 and it was the earliest thermomechanically processed NiTi alloy without superelastic characteristics whether at room or body temperature.(7) CM wire can be distorted due to reorientation of the martensite variation due to changing phase composition.(46) As a result, unlike austenitic NiTi files, CM wire devices do not entirely adjust when curved root canals are prepared. When compared to RevoS, ProTaper Next and Reciproc files, the use of Hyflex CM files led in much less root canal straightening during preparation (5).

DSC study demonstrated that the austenite final temperature of CM wire devices is higher than the temperatures observed within root canals (approximately 4755°C) (41). It was also discovered that unutilized Hyflex CM devices had Af temperatures of roughly 3237°C, but utilized Hyflex

CM instruments had Af temperature of approximately 5461°C.(43) Although, XRD study of Hyflex CM and Typhoon CM (Clinician's Choice Dental Products, New Milford, CT, USA) found that at room temperature, both fresh and second hand CM wire devices had a combination of austenite and martensite structures, with a little amount of Rphase.(47) CM wire instruments were more flexible than M₂ wire and

the standard NiTi devices (2). The primary reason for this enhanced flexibility is because the critical stress needed to cause martensite reorientation (from twinned to deformed martensite) in martensitic instruments is substantially lower than the required stress needed to cause stress-induced martensite (SIM) transition (from austenite to deformed martensite) in austenitic devices (Fig. 4) (18). Even after having enhanced flexibility that might result in a negative impact on cutting efficiency, Hyflex CM instruments outperformed electropolished and conventional NiTi instruments (9).

CM wire instruments demonstrated much greater cycle fatigue resistance than Mwire and standard SE NiTi instruments, possibly due to the martensitic condition (24). CM wire instruments also demonstrated a larger deflection angle at failure than Mwire and traditional SE NiTi, although having nearly a same optimum torque (18).



Figure 4. Schematic representation of phase crystallographic transformation of NiTi alloy induced by stress or temperature

Gold and Blue heat-treated instruments

Dentsply Tulsa Dental (Tulsa, OK, USA) launched the ProFile VortexBlue files, the earliest endodontic instruments with a distinctive colour of blue (41). There are two major colour of thermal-treated NiTi systems as Gold and Blue (48,49). ProFile VortexBlue and ProTaper Gold (Dentsply Sirona Endodontics) are the representative rotary systems and Reciproc Blue (VDW) and WaveOne Gold (Dentsply Sirona Endodontics) are the representative reciprocating systems (49). These devices can also be deformed and have a controlled memory effect (24). The primary distinction of CM wire and the Gold and Blue thermal treated devices is that the former are crushed prior to undergoing a patented post-machining thermal treatment (50).

The titanium oxide coating that remains after the post-machining heat treatment is the reason of the remarkable blue colour on the surface of Vortex Blue instruments (51).

Vortex Blue's austenite finish temperature was close to body temperature (38.5°C), whereas the martensite initial temperature is around 31°C (52). The Vickers surface hardness of the blue heat-treated devices is softer than that of the Mwire instruments (53). The blue heat-treated file's regulated memory behaviour, independent of the lower transformation temperatures, shows that these instruments contain more steady martensite than M-wire, resulting in a smoother and more durable NiTi alloy (17).

The distinctive colour of the gold heat-treated instruments may also be caused by the upper layer (25). DSC examination of ProTaper Gold found that the austenite finish temperature was roughly 50°C, indicating that when utilized at clinical temperatures, these instruments primarily contain martensite or Rphase (48,50). Due to their martensitic condition, all Gold and Blue thermal-treated files demonstrated greater flexibility and fatigue endurance as-

compared to standard SE NiTi and Mwire instruments (54). Hyflex EDM files is the only one that outperformed ProTaper Gold, WaveOne Gold, and Reciproc Blue regarding cyclic fatigue tolerance (51). According to Kaval et al., Compared to Hyflex EDM and ProTaper Universal, ProTaper Gold featured a much greater maximum torque, but Hyflex EDM is the one with a higher deformation angle (50). ProTaper Gold outperformed ProTaper Universal in lateral cutting motion (48).

MaxWire

MaxWire was introduced as a new patented thermomechanically treated NiTi alloy (Martensite-AusteniteelectropolishfileX) by FKG Dentaire Manufacturer. This is the earliest instrument to combine shape memory capacity with SE characteristics (55). The XPEndo Shaper and the XPEndo Finisher are two MaxWire instruments that are commercially available (21). While these instruments are reasonably straight in the martensitic state at ambient temperature, when subjected to intracanal temperatures, they curve due to transition to the austenitic state (56). As a result, when placed inside the root canal (martensiticphase to austeniticphase), they have a form memory effect and are superelastic during canal preparation (25). Because of the curve shape, root canals with complex morphology can be created with the file that adapts to the canal abnormalities (57).

The cycle fatigue endurance of the XPEndo Shaper was much greater compared to Hyflex CM, VortexBlue, and iRaCe, however it had less torsional resistance than Vortex (23). It is worth noting that this instrument's taper of 0.01mm/mm had an effect on its cyclic and torsional resistance.(41) This is most likely due to the smaller diameter improving the resistance to cycle fatigue and decreasing the torsional strength (58).

Electrical discharge machining (Hyflex EDM)

The Hyflex EDM was the earliest instrument made using an electrical discharge machining (EDM) method (Fig. 4) (59). According to the manufacturer, this technique hardens the skin of the instrument, leading to greater fracture resistance and better cutting performance (60). EDM is a representative noncontact machining technique that uses a pulsed electrical discharge to remove material accurately (13). The workpiece and the cutting tool (electrode) must both be good electrical conductors for EDM to work (17). A dielectric liquid is used to embed the machining tool, which is then moved closer to the workpiece until the gap is narrow enough for the input current to ionize the dielectric fluid

(13). Fine particles from the workpiece are vaporized by the ensuing spark, which causes them to re-solidify in the dielectric liquid and be successively removed. EDM, as opposed to the traditional grinding, does not need to touch the work item directly, hence eliminating the chances of inducing mechanical stress (61).

Optically observed metallographic study of Hyflex EDM instruments discovered a molecular structure dominated by translucent grains (presumably martensite), that alternate with big flat grains (assumed to be austenite) (26). In contrary, XRD analysis found that Hyflex EDM is made of martensite and significant quantities of Rphase, whereas Hyflex CM is a mix of martensite and austenite (62).

When contrasted with Hyflex CM, Mwire, and standard SE NiTi devices, Hyflex EDM exhibited much higher cyclic fatigue resistance (14). Hyflex EDM's flexibility is comparable to that

of other CM wire instruments. It has been observed that Hyflex EDM can generate a centralized root canal preparation (29). Furthermore, when compared to Mwire files, Hyflex EDM files had a higher rotation angle at fracture, but it had a lesser torque to fracture (63). Despite the lower austenite phase, Hyflex EDM instruments were harder than conventionally manufactured CM wire files, resulting to the surface hardening process used to make EDM files (10).

Conclusion

Significant changes in the metallurgical characteristics of NiTi alloys have been found as a result of thermomechanical heat treatments. Endodontic treatment quality was improved by the current modifications in the production process of NiTi alloys, which ensures that modern endodontic instruments have better mechanical properties. The pre- and post-machining thermo mechanical heat treatment approach improved instrument lifetime by enhancing resistance to flexural and cyclic fatigue. In order to increase the quality, safety, and effectiveness of endodontic devices and, ultimately, the clinical success rates, deeper study is required into the effects of the heat treatment technique for re treatment files.

AUTHOR CONTRIBUTIONS

DD, TA, HCK, PVA contributed to data acquisition, analysis, and interpretation and drafted the manuscript; All authors contributed to conception and design and critically revised the manuscript. All authors gave final approval and agreed to be accountable for all aspects of the work.

CONFLICT OF INTEREST DECLARATIONS

Declarations of interest: none

References

- Walia H, Brantley WA, Gerstein H. An initial investigation of the bending and torsional properties of nitinol root canal files. *J Endod* 1988; 14: 346-51.
- Zhou H, Peng B, Zheng YF. An overview of the mechanical properties of nickel-titanium endodontic instruments. *Endod Top* 2013; 29: 42-54.
- Harrison T. Cyclic fatigue resistance of nickel-titanium rotary files in the martensitic state: a systematic review. *SYSTEMATIC REVIEW OF CYCLIC FATIGUE RESISTANCE INMARTENSITIC NITI FILES Cyclic fatigue resistance of nickel-titanium rotary files in the martensitic*. 2019; Available from: <http://hdl.handle.net/10034/623368>
- Dennis D, Farahanny W, Prasetya W, Batubara FY. Biological and mechanical principles of chemomechanical preparation in root canal therapy: A review. *Int J Clin Dent* 2021; 14: 187-98.
- Journal W, Pharmaceutical OF. Niti Technology Has Revolutionised Endodontic Therapy With Newer Thermo mechanically Treated Niti Alloys : a Literature.2021;7(6):148-58.
- Jo HJ, Kwak SW, Kim HC, Kim SK, Ha JH. Torsional resistance of heat treated nickel-titanium instruments under different temperature conditions. *Materials (Basel)*. 2021; 14: 1-10.
- Bansode Wavdhane Seema D, Pathak PM, Khedgikar DB, Rana H. Evolution of Rotary NITI File Systems: A

Literature Review Endodontic. 2016;(December):91–4.

8. Tabassum S, Zafar K, Umer F. Nickel titanium rotary file systems: What's new? *Eur Endod J*. 2019;4(3):111–7.
9. Zupanc J, VahdatPajouh N, Schäfer E. New thermomechanically treated NiTi alloys – a review. *Int Endod J*. 2018;51(10):1088–103.
10. Krishnan V, Nair RS, Ashok L, Angelo MC. An Overview of Thermomechanically HeattreatedNickel–Titanium Alloy Used in Endodontics. *Conserv Dent Endod J*. 2019;4(2):34–8.
11. Duerig TW, Bhattacharya K. The Influence of the RPhase on the Superelastic Behavior of NiTi.Shape Mem Superelasticity. 2015;1(2):153–61.
12. Martins JNR, Silva EJNL, Marques D, Pereira MR, Vieira VTL, ArantesOliveira S, et al. Design, Metallurgical Features, and Mechanical Behaviour of NiTi Endodontic Instruments from Five Different HeatTreated Rotary Systems. *Materials (Basel)*. 2022;15(3).
13. Martins SCS, Silva JD, Viana ACD, Buono VTL, Santos LA. Effects of heat treatment and design on mechanical responses of NiTi endodontic instruments: A finite element analysis. *Mater Res*. 2020;23(3):26–8.
14. Aoun C, Nehme W, Naaman A, Khalil I. Review and classification of endodontic heat treatment procedures. *Int J Curr Res*. 2017;(June).
15. M HJ, Chakravarthy D, S V. Metallurgy of Rotary Files- A Review. *J Curr Med Res Opin*. 2021;04(6):975–80.
16. Pillay M, Vorster M, Van der Vyver PJ. Fracture of endodontic instruments - Part 1: Literature review on factors that influence instrument breakage. *South African Dent J*. 2020;75(10):553–63.
17. Pereira ESJ, Peixoto IFC, Viana ACD, Oliveira II, Gonzalez BM, Buono VTL, et al. Physical and mechanical properties of a thermo mechanically treated NiTi wire used in the manufacture of rotary endodontic instruments. *Int Endod J*. 2012;45(5):469–74.
18. Thompson SA. An overview of nickel titanium alloys used in dentistry. *Int Endod J*. 2000;33(4):297–310.
19. Grande NM, Plotino G, Butti A, Buono L. Modern Endodontic NiTi Systems : Race. 2017;5(1):3–8.
20. Martins JNR, DiBernardo J. Torsional failure characteristics of a NiTi file based on a case report. *Rev Port Estomatol Med Dent e Cir Maxilofac* [Internet]. 2010;51(2):85–9. Available from: [http://dx.doi.org/10.1016/S16462890\(10\)700911](http://dx.doi.org/10.1016/S16462890(10)700911)
21. Soni MR, Hegde S, Mathew S, Madhu KS. Rotary systems: An insight. *J Dent Orofacial Res*. 2014;10(2):16–22.
22. Gandhi C, Gupta S, Gupta P. Niti : A Savior in Endodontics. 2021;8(11):2020–2.
23. Gu Y, Kum KY, Perinpanayagam H, Kim C, Kum DJ, Lim SM, et al. Various heattreated nickel–titanium rotary instruments evaluated in Sshaped simulated resin canals. *J Dent Sci*. 2017;12(1):14–20.
24. Ounsi HF, Nassif W, Grandini S, Salameh Z, Neelakantan P, Anil S. Evolution of nickel titanium alloys in endodontics. *J Contemp Dent Pract*. 2017;18(11):1090–6.
25. Liang Y, Yue L. Evolution and development: enginedriven endodontic rotary nickel titanium instruments. *Int J OralSci*. 2022;14(1):1–8.
26. Zanza A, Seracchiani M, Reda R, Di Nardo D, Gambarini G, Testarelli L. Role of the crystallographic phase of NiTi rotary instruments in determining their torsional resistance during different bending conditions. *Materials (Basel)*. 2021;14(21).
27. Pedullà E, Lo Savio F, La Rosa GRM, Miccoli G, Bruno E, Rapisarda S, et al. Cyclic fatigue resistance, torsional resistance, and metallurgical characteristics of M3 Rotary and M3 Pro Gold NiTi files. *Restor Dent Endod*. 2018;43(2):1–10.
28. Vieira TM, Cardoso RM, Alves NCC, Emanuel Acioly Conrado De Menezes S, Batista SM, Silva SDA, et al. Cyclic Fatigue Resistance of Blue HeatTreated Instruments at Different Temperatures. *Int J Biomater*. 2021;2021.
29. Aminsohanni M, Khalatbari MS, Meraji N, Ghorbanzadeh A, Sadri E. Evaluation of the fractured surface of five endodontic rotary instruments: A metallurgical study. *Iran Endod J*. 2016;11(4):286–92.
30. Li H, Zhao J ping, Wang Z yu, Ding L. Effect of heat treatment on cyclic deformation properties of Fe–26Mn–10Al–C steel. *J Iron Steel Res Int*. 2019;26(2):200–10.
31. Kwak SW, Shen Y, Liu H, Wang Z, Kim HC, Haapasalo M. Heat Treatment and Surface Treatment of Nickel–Titanium Endodontic Instruments. *Front Dent Med*. 2021;2(October):1–6.
32. Zafar MS. Impact of Endodontic Instrumentation on Surface Roughness of Various Nickel- Titanium Rotary Files. *Eur J Dent*. 2021;15(2):273–80.
33. Islambasic M, Coelho MS, Pettiette MT, Tawil PZ. Nominal size and taper analysis of novel metallurgy NiTi files. *Eur Endod J*. 2016;1(1).
34. Oktavia E, Abidin T, Dennis D. Effect of sodium hypochlorite, EDTA, and chitosan solution on corrosion and quantity of extruded nickel ions using two rotary instruments (In Vitro). *World J Dent*. 2019;10(3):207–13.
35. Generali L, Malovo A, Bolelli G, Borghi A, Rita G, La M, et al. New Green NiTi Reciprocating Instruments. 2020;
36. TanomaruFilho M, Espir CG, Vençao AC, MacedoSerrano N, CamiloPinto J, Guerreiro- Tanomaru JM. Cyclic fatigue resistance of heattreated nickeltitanium instruments. *Iran Endod J*. 2018;13(3):312–7.
37. Mahmud AS, Ng CW, Razali MF. Effect of surface oxidation on shape memory behaviour of NiTi alloy. *AIP Conf Proc*. 2016;1774(October 2016):1–7.
38. Shen Y, Zhou HM, Zheng YF, Peng B, Haapasalo M. Current challenges and concepts of the thermomechanical treatment of nickelitanium instruments. *J Endod*. 2013;39(2):163–72.
39. Lopes WSP, Vieira VTL, Silva EJNL, Dias PRN, Lopes HP, Elias CN, et al. Mechanical properties of reciprocating thermally treated NiTi endodontic instruments / Propriedades mecânicas de instrumentos endodônticos de NiTi reciprocantes tratados termicamente. *Brazilian J Dev*. 2021;7(9):88149–62.
40. Kataia EM, Nagy MM, Kataia MM, Khalil HF. Shaping ability of two heat treated rotary NiTi instruments using different kinematics/in vitro study. *Bull Natl Res Cent* [Internet]. 2021;45(1). Available from: <https://doi.org/10.1186/s4226902100499w>
41. Braz Fernandes FM, Alves AR, Machado A, Oliveira JP. Effect of heat treatments on NiTi endodontic files. *Cienc e Tecnol dos Mater*. 2017;29(1):e15–8.
42. Ye J, Gao Y. Metallurgical characterization of MWire nickel titanium shape memory alloy used for endodonticrotary instruments during lowcycle fatigue. *J Endod* [Internet]. 2012;38(1):105–7. Available from: <http://dx.doi.org/10.1016/j.joen.2011.09.028>
43. Alsofi L, Rajkhan W, AlHabib M, Ashe H, Alnowailaty Y, Balto K. Characterization of the differential efficacy of austenitic vs martensitic NiTi rotary files in nonsurgical

root canal retreatment: A microCT analysis. *Front Biosci - Landmark*. 2021;26(9):465–74.

44. Alizzio D, Savio F Lo, Bonfanti M. Numerical and experimental analysis in endodontic rotary files under cyclic fatigue or torsional stress. *CEUR Workshop Proc*. 2020;2768:59–65.

45. R N. NiTi Endodontics: Contemporary Views Reviewed. *Austin J Dent*. 2018;5(4).

46. Simpura J. Research Article. *Nord Stud Alcohol Drugs*. 1998;15(3):131–2.

47. Galal M. Metallurgical effect on the mechanical behavior of rotary endodontic files using finite element analysis. *Bull Natl Res Cent*. 2019;43(1):0–4.

48. Alqedairi A, Alfawaz H, Abualjadayel B, Alanazi M, Alkhalfah A, Jamleh A. Torsional resistance of three ProTaper rotary systems. *BMC Oral Health*. 2019;19(1):1–6.

49. Shubhashini N, Sahu GK, Consul S, Nandakishore K, Idris M. Rotary Endodontics or Reciprocating Endodontics: Which is New and Which is True? *J Heal Sci Res*. 2016;7(2):51–7.

50. 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(12):1840–3.

51. Vilaverde Correia S, Nogueira MT, Silva RJC, Pires Lopes L, Braz Fernandes FM. Phase Transformations in NiTi Endodontic Files and Fatigue Resistance. 2009;07004:0–6.

52. Bansal S, Taneja S, Kumari M, Dhillon M. Comparative evaluation of the shaping ability of rotary systems of varying metallurgy in curved canals and its analysis using conebeam computed tomography: An in vitro study. *Endodontontology*. 2019;31(2):158.

53. Radwański M, Łęski M, Pawlicka H. The influence of the manufacturing process of rotary files on the shaping of Lshaped canals. *Dent Med Probl*. 2018;55(4):389–94.

54. Madarati AA, Habib AA. Modalities of using endodontic nickel-titanium rotary instruments and factors influencing their implementation in dental practice. *BMC Oral Health*. 2018;18(1):1–10.

55. Gavini G, dos Santos M, Caldeira CL, Machado ME de L, Freire LG, Iglesias EF, et al. Nickel-titanium instruments in endodontics: A concise review of the state of the art. *Braz Oral Res*. 2018;32:44–65.

56. Haapasalo M, Shen Y. Evolution of nickel-titanium instruments: from past to future. *Endod Top*. 2013;29(1):3–17.

57. Zanza A, D'angelo M, Reda R, Gambarini G, Testarelli L, Di Nardo D. An update on nickel-titanium rotary instruments in endodontics: Mechanical characteristics, testing and future perspective—An overview. *Bioengineering*. 2021;8(12).

58. Dobrzański LA, Dobrzański LB, Dobrzańska Danikiewicz AD, Dobrzańska J. Nitinol Type Alloys General Characteristics and Applications in Endodontics. *Processes*. 2022;10(1).

59. Papworth B, Papworth B. The Evaluation of the Cutting Efficiency of Nickel-Titanium Rotary Files : A New InVitro Dentin Model The Evaluation of the Cutting Efficiency of Nickel-A New InVitro Dentin Model. 2002;(June).

60. Khabadze Z, Mordanov O, Balashova M, Stolov L, Panagryyan A, Bokova R, et al. Laboratory Rational of Changes in the Crystal Lattice of Nickel-Titanium Endodontic Rotary Files in Autoclaving. *Int J Dent*. 2020;2020:12–5.

61. RuizSánchez C, FausMatoses V, AlegreDomingo T, FausMatoses I, FausLlácer VJ. An in vitro cyclic fatigue resistance comparison of conventional and new generation nickel-titanium rotary files. *J Clin Exp Dent*. 2018;10(8):e805–9.

62. Sung HJ, Ha JH, Kim SK. Influence of taper on the screw-in effect of nickel-titanium rotary files in simulated resin root canal. *J Korean Acad Conserv Dent*. 2010;35(5):380.

63. Zhou HM, Shen Y, Zheng W, Li L, Zheng YF, Haapasalo M. Mechanical properties of controlled memory and superelastic nickel-titanium wires used in the manufacture of rotary endodontic instruments. *J Endod* [Internet]. 2012;38(11):1535–40. Available from: <http://dx.doi.org/10.1016/j.joen.2012.07.006>