



## Nickel-titanium files in endodontics: development, improvement, and modifications of nickel-titanium alloy

Nikl-titanijumski instrumenti u endodonciji: razvoj, usavršavanje i modifikacije nikl-titanijumskih legura

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### Introduction

Numerous innovations in endodontic instrumentation were introduced in the early 1990s, and the concept of developing flexible endodontic files was significantly improved using nickel-titanium (NiTi) alloy instead of long-used stainless steel. The introduction of “smart” NiTi alloy is one of the biggest evolutionary shifts in endodontics because it has significantly improved the quality and efficiency of canal processing, with a significant reduction in complications and errors<sup>1-4</sup>.

Civian et al.<sup>5</sup> were the first to suggest the use of NiTi alloys for the production of endodontic files in 1975, and Walia et al.<sup>6</sup> were the first to introduce hand-held NiTi endodontic files made of treated orthodontic wire. The first NiTi rotary files with a 0.002 cone (introduced in 1992) were designed by Dr. John McSpadden<sup>1</sup>.

Complications during instrumentation related to the inherent stiffness of steel files have been significantly reduced thanks to the specific characteristics of the innovative NiTi alloy. Superelasticity (SE) and shape memory (SM) are the results of the phase transformation of the NiTi alloy, i.e., solid phase changes and transition from austenitic (parent phase) to martensitic phase<sup>7</sup>.

Numerous sets of rotary NiTi files have been introduced during the last few decades (there are currently more than 160 of them), and new ones appear every day<sup>1</sup>. The development of machine files is chronologically classified into

five generations, with a tendency to improve their individual performance and a maximum reduction of possible shortcomings<sup>8,9</sup>.

The focus of the endodontic dental industry was initially aimed at increasing the cutting efficiency of the files, i.e., reducing complications (fractures), during canal treatment. Modern generations primarily include different designs of the working part of the file related to cross-section, fixed or variable conicity, number and appearance of blades, and arrangement of blades along the active part, i.e., the presence or absence of radial surfaces<sup>1,7,8</sup>.

However, despite different design solutions, the occurrence of deformations and sudden fractures during instrumentation was not excluded, so technological solutions related to the modification and transformation of the basic NiTi alloy followed. In addition to changes related to surface and heat treatments of NiTi alloys, the number of files required for instrumentation has been reduced, and a new concept of activating machine files has been introduced to prevent the deformation and breakage of files<sup>8,10-13</sup>. It has been confirmed that different production processes, design solutions, but also the physical properties of the alloy, affect the clinical performance of machine files, i.e., their efficiency and safety during instrumentation<sup>8</sup>. The focus was, therefore, shifted towards the application of various technologies of heat and mechanical treatment of NiTi alloy in order to optimize the microstructure and thus increase the flexibility of endodontic files<sup>11,14</sup>.

Modern technological processes of the development of machine files are based on the knowledge that the mechanical properties of equiatomic NiTi alloy depend on the phase transformation caused by stress and the fact that these microstructural changes can be controlled by thermomechanical treatments<sup>14</sup>. It has also been confirmed that the metallurgical characteristics of NiTi alloy (composition, microstructure, and phase constitution) significantly affect the basic performance of files<sup>11, 15, 16</sup>.

The aim of this paper was to present the development, improvement, and modification of NiTi alloy and indicate how the effects of different thermomechanical alloy treatments and specific surface and heat treatments of files affect the improvement of clinical performance and safer and more efficient root canal cleaning and shaping.

### Crystallographic structure of NiTi alloys

The equiatomic NiTi alloy consists of approximately 56 percent by weight (wt%) of Ni and 44 wt% of Ti. The difference in weight percentages is due to the different molecular weights of these two elements (Ni 58.69 g/mol and Ti 47.87 g/mol)<sup>17</sup>. The specifics of alloys are the direct and strong bonds between electrons, which are responsible for their exceptional characteristics, such as SE and SM effect (SME). The NiTi alloy has three different phase microstructures that are temperature dependent: austenite, martensite, and a transient, R-phase. The austenitic phase gives a strong and hard form, and the martensitic and R-phase a softer form of the alloy that is easily deformed<sup>18</sup>. All three phases of transformation affect the mechanical characteristics of the alloy, and the change of phase in the solid state is responsible for the elastic properties, i.e., the martensitic transformation induced mainly by stress or temperature reduction<sup>1</sup>. After elastic or pseudoplastic deformation, the alloy undergoes a thermoelastic transformation from the austenitic to the martensitic phase. Quite a small force is enough to cause the alloy to bend at this stage. The whole structure recovers after the cessation of stress, returning to the austenitic phase, where it takes on its original shape (stress-induced thermoelastic transformation). The martensitic phase is generated by the stress of the material in the austenitic state and allows high stresses<sup>17</sup>. Atoms in the martensitic transformation are rearranged into a new, more stable crystal structure without changing the chemical composition of the matrix but with a macroscopic change in the shape of the material. This phase transformation occurs by transitioning the high-temperature, spatially centered cubic or tesserall austenitic, parent phase, into a rhombic or monoclinic martensitic phase. The martensitic transformation occurs due to shear forces and most often passes through the middle tetragonal phase (R-phase), where the martensitic areas have the same crystal structure but a different spatial orientation. By heating the material in the martensitic phase, a reversible transformation occurs, and martensite returns to the austenitic phase<sup>17</sup>.

A specific property of SM is the ability of the alloy to completely regain its original shape when heated above the temperature of transformation of martensite into austenite

(temperature varies depending on the chemical composition of the alloy). Among various metal alloys that show SE and SM, NiTi has the best biocompatibility and exceptional corrosion resistance due to the existence of a surface layer of Ti oxide<sup>19</sup>. Although only one manufacturer (Dentsply, Maillefer Instruments SA, Ballaigues, Switzerland) has published the absolute composition of the conventional alloy (56 wt% of Ni and 44 wt% of Ti) and the detailed technological process of producing rotary files, it is considered the best ratio that provides superelastic properties<sup>20</sup>.

The dominance of the desired properties of the files (superelastic or more pronounced SME) is enabled by the variation of the composition of the NiTi alloy. Increasing the proportion of Ni or its replacement by trace elements, e.g., cobalt (less than 2% by weight), results in a reduced phase transformation temperature, while an increase in temperature (e.g., annealing) also increases the phase transformation temperature<sup>7</sup>. The temperature transformation of NiTi alloys depends on the volume ratio of Ni and Ti and can range from -50 °C to +100 °C<sup>7</sup>. It has been confirmed that even a change of 0.1% in the composition of the alloy can lead to a change in the transformation temperature of 10 °C, which can subsequently affect its mechanical characteristics<sup>21</sup>. Otsuka and Ren<sup>19</sup> found that increasing the Ni content leads to a drastic decrease in the transformation temperature, so the focus of the development of new NiTi files is directed toward Ni-rich alloys.

### Thermo-mechanical treatments of NiTi alloy

Thermo-mechanical treatment is a metallurgical process that involves combining mechanical (compression or forging, rolling, drawing, etc.) and heat treatment (water quenching, heating, and cooling) methods of alloy processing in a single process<sup>1, 20-22</sup>. Thermo-mechanical treatments can be applied during the production process of the alloy and files, i.e., subsequent treatment of finished files.

Heat treatment of alloy is a modern approach to the regulation of increasing resistance to cyclic fatigue of endodontic files. This process consists of heating the material to a certain temperature and cooling it after a certain time under controlled conditions<sup>14, 20</sup>. It has been confirmed that temperature, i.e., the heating time and the cooling rate during alloy production, affect the SE and SM of NiTi files<sup>1</sup>.

New endodontic files with superior mechanical properties have been developed by the thermo-mechanical treatment that goes through the processes of aging, annealing, and recrystallization of the alloy<sup>1</sup>. Heat treatment leads to four different reactions: a) changes in chemical composition, b) recovery of defects, c) reduction of defects by recrystallization, and d) transformations of the structural phase<sup>1</sup>.

Aging gives the alloy greater mechanical strength and implies even heating of the alloy to about 500 °C and then rapid cooling (usually in water) because this prevents the deposition of alloying elements. Hardened parts are removed by annealing (heating to 300 °C–500 °C), and the alloy is then slowly cooled. Recrystallization implies a cold deformed structure with a changed set of grains, which reduces the hardness of the alloy and increases its forgeability<sup>1</sup>.

Heat treatment maintains the crystallographic structure of the alloy and provides files with greater flexibility and fracture resistance<sup>23, 24</sup>. NiTi endodontic files mainly contain an austenite phase (conventional NiTi, M-wire, R-phase) or a martensitic phase [controlled memory (CM) wire and gold and blue heat-treated NiTi alloys]<sup>23, 25</sup>. Superelastic alloys with a stable martensitic phase, which shows a lower modulus of elasticity (30–40 GPa) compared to austenitic (80–90 GPa), have also been developed by special heat treatment, while this modulus in the R-phase is lower than in martensite<sup>25</sup>. The mechanical properties of the alloy also depend on the ambient temperature: if the temperature is above the austenite finish (austenitic state), the alloy becomes hard, with superior superelastic properties; if the temperature is below the martensite finish (martensite state), it becomes soft and ductile and deforms easily with SME. The martensitic phase provides superior resistance to cyclic fatigue compared to austenitic due to the reorientation of the double phase structure<sup>24, 26, 27</sup>.

### Thermal preparation of NiTi alloy

Conventional NiTi wires are formed by cold drawing, resulting in a microstructure containing martensite residues in the austenitic matrix. Internal stresses can be released, and the disadvantages of rearrangement of the crystal lattice can be reduced by heat treatment of such an alloy (450 °C–550 °C)<sup>1, 28, 29</sup>.

### M-wire

The M-wire, characterized by an austenitic phase with a small proportion of stable R- and martensitic phase<sup>11, 14</sup>, has contributed to the development of superelastic characteristics of NiTi endodontic files. This wire was introduced (2007) by Sports Wire LLC (Langley, OK, USA) and Dentsply Tulsa (Dental Specialties, Tulsa, OK, USA) and proposed for use in endodontics<sup>24–26</sup>.

The M-wire is composed of Nitinol 508 (Ni 55.8%), which is subjected to specific heat treatments at different temperatures. This type of alloy contains areas in the deformed martensitic phase, premartensitic R-phase, but also in austenitic phase. The finishing of the wire in the austenitic phase ranges from 45 °C to 50 °C, maintaining a pseudoelastic state<sup>20</sup>. Unlike the austenitic form, where the alloy is resistant and hard, the M-wire in the martensitic phase becomes softer, more flexible, and easily deformed, with a significantly lower possibility of stress fracture<sup>11</sup>. Martensitic phase transformation also has excellent resistance to fatigue due to its ability to absorb energy<sup>20</sup>.

Compared to files produced from conventional NiTi alloys, files made of M-wire have higher resistance to cyclic fatigue and improved mechanical properties<sup>14, 24, 26</sup>.

Files made of M-wire are ProFile GTKS, ProFile Vortex, ProTaper Next, Path Files (Dentsply Sirona, York, PA, USA), and WaveOne (Dentsply Maillefer, Ballaigues, Switzerland) and Reciproc (VDW, Munich, Germany). De Vasconcelos et al.<sup>30</sup> found that increased alloy flexibility and

increased fatigue resistance (martensitic phase) also provide a better arrangement of the crystal structure. Gao et al.<sup>31</sup> explained the higher resistance to cyclic fatigue of files made of M-wire by the lower appearance of cracks due to the better crystal orientation of the martensitic phase.

### R-wire

During the production process, a conventional NiTi alloy in the austenitic phase is transformed into a rhombohedral crystal structure, with the formation of the middle R-phase (the form between austenite and martensite)<sup>11, 19</sup>.

It is formed during the direct transformation of martensite into austenite (due to heating) or during the inverse transformation from austenite to martensite (by cooling) and shows a lower modulus of elasticity compared to the martensite and austenitic phases. Files made of R-phase alloy have a complete austenitic form at ambient and body temperature and allow greater flexibility and increased resistance to cyclic fatigue<sup>14, 20</sup>. In contrast, Park et al.<sup>32</sup> have observed that this production process does not affect the increased resistance to torsional fractures of files.

R-wire endodontic files include Twisted and K3 systems. SironEndo (Orange, CA, USA) developed the Twisted File system in 2008 and the Twisted File Adaptive in 2013 using the production process that involved transforming raw austenitic NiTi wire by thermal process into R-phase, twisting the wire and then conditioning its surface<sup>14, 25</sup>.

Ghabbani's profilometric studies confirm the lower occurrence of surface deformations after instrumentation on the Twisted File system compared to M-wire files (ProTaper Next, Dentsply Maillefer, Ballaigues, Switzerland)<sup>33</sup>.

SironEndo developed K3KSF (SironEndo Orange, CA, USA) in 2011, which is a system that takes advantage of R-phase technology and represents an advanced version of the files, as it is subjected to a specific heat treatment<sup>14</sup>. These files are made by the micro-milling process and then subjected to a special heat treatment that significantly improves flexibility and strength, with modification of the crystal structure of the alloy<sup>14</sup>. Heat treatment can change the temperature of the phase transformation by repairing the crystal lattice defects and reducing the internal deformation energy. K3KSF files are a new evolutionary variant of the K3 system (created in 2011), and the R-wire provides them with better mechanical properties compared to files produced by a traditional process<sup>1, 34</sup>. Unlike Twisted File and Twisted File Adaptive, produced by plastic deformation, the innovative way of production involves twisting metal wire and its heat treatment by recrystallization and provides the Twisted File system with greater elasticity and resistance to cyclic fatigue<sup>25</sup>. The protected production process ensures greater integrity of the crystal lattice structure of the metal by twisting and significantly increases the resistance of the files to breakage<sup>1</sup>. Increased flexibility in R-phase files allows better centering along the canal and a lower possibility of transport compared to conventional NiTi systems, while their resistance to cyclic fatigue is similar to files made of M-wire<sup>25, 35</sup>.

### Alloy with controlled memory wire

The NiTi wire with CM-wire was introduced in 2010 and represents the first thermomechanically treated alloy that does not possess superelastic properties, neither at room nor at body temperature<sup>25,36</sup>. This wire is made of Nitinol SE508 alloy, with a lower Ni mass percentage (52%), compared to conventional SE alloys (54%–57%)<sup>1,14</sup>. The protected thermal process of CM-alloy formation involves heating and cooling in order to obtain a stable martensite phase at body temperature and thus better mechanical properties (increased flexibility, reduced SM, and increased transformation temperature)<sup>1,25,33</sup>.

Recent studies have shown that the formation temperature of the austenitic phase in files made of CM alloy is about 47 °C when the composite of martensitic phase, R-phase, and austenitic phase is achieved at room temperature<sup>1,25,37</sup>. This alloy allows the files to be bent before application into the canal, which significantly increases their resistance to cyclic fatigue. Files made of CM-wire are returned to their original state after sterilization and can be reused until the moment of irreversible deformation when they should be discarded<sup>1,17,25,37</sup>. These files show increased flexibility and better centering along the canal<sup>1,17,25</sup>.

Endodontic files made of CM alloy (CM-wire) include Hyflex CM, Typhoon Infinite Flex, and Pro Design systems. Coltene/Whaledent (Cuyahoga Falls, OH) introduced Hyflex CM files made of this alloy in 2011. These files show greater resistance to cyclic fatigue and are subjected to plastic deformation during use but return to their initial state after autoclaving<sup>1,15,17</sup>. Shen et al.<sup>38</sup> pointed out that HyFlex CM files show three to eight times higher resistance to cyclic fatigue compared to files made of conventional NiTi wires. Despite their lower tensile strength (1094 MPa vs. 1415 MPa in conventional files), CM files are more resistant to deformation (58.4%–84.7%) compared to the conventional ones (16.7%–27.5%), which indicates their superior flexible properties<sup>1</sup>. The transport rate of HyFlex CM files is similar to other super-elastic NiTi files, but canal correction is significantly reduced compared to Revo-S (Micro-Mega, Besançon, France), ProTaper Next (Dentsply Sirona Endodontics, Ballaigues, Switzerland), and Reciproc files (VDW, Munich, Germany)<sup>9,23</sup>.

Typhoon Infinite Flex files (Clinician's Choice Dental Products, New Milford, CT, USA) were created in 2011; they show 150% higher resistance to cyclic fatigue than files made of M-wire and 390% compared to those of conventional NiTi alloy<sup>1</sup>.

ProDesign R and ProDesign Logic systems (Easy Dental Equipment, Belo Horizonte, MG, Brazil) were created in 2014; they have an S-shaped cross-section, an inactive tip, and variable spiral angles with two blades. These systems are designed with a single file concept and differ only in the way the files are activated (ProDesign Logic is used in full rotation, while ProDesign R is designed for reciprocal activation)<sup>1</sup>.

### Max-wire

FKG (FKG Dentaire, La-Chaux-de-Fonds, Switzerland) developed a special NiTi alloy Max-wire (Martensite-Austenite Electropolishing-Flex, FKG) for the production of XP systems (XP-endo Shaper, XP-endo Finisher, XP-endo Retreatment) in 2015<sup>1</sup>. The transition from martensitic to austenitic phase occurs during the processing of the alloy at temperatures equal to or higher than 35 °C. These files are relatively straight at room temperature (M-phase, martensitic state), but due to the exposure to intracanal temperature and phase transformation, they change into another, bent form (A-phase, austenitic state). The transition from the martensite phase to the austenite phase occurs naturally at body temperature between 32 °C and 37 °C (austenite temperature around 35 °C).

XP-endo Shaper with a diameter of #30 size and a variable cone (0.01–0.04) and XP-endo Finisher with a diameter of #25 size and a conicity of 0.00 are, with eccentric rotation, able to adapt to the morphology of the root canal system, expanding or contracting as they progress along the canal<sup>1,39,40</sup>.

Leoni et al.<sup>41</sup> pointed to more efficient cleaning of the XP-endo Finisher dentinal walls compared to passive ultrasonic irrigation, while Keskin et al.<sup>42</sup> confirmed that XP-endo Finisher removes calcium hydroxide residues from the canal more effectively in combination with passive ultrasonic irrigation. Živković et al.<sup>39,40</sup> confirmed efficient cleaning of the apical part of the canal using XP-endo Finisher files.

### T-wire

The new martensitic alloy heat treatment system is known as T-wire technology, which, according to the manufacturer, significantly increases the resistance of files to cyclic fatigue (up to 40%)<sup>1</sup>.

Endodontic files made of NiTi alloy (T-wire) are One Endo and Exo Endo, or TwoShape system. The design of these files was created by combining two or more different cones on the same file. The files have a specific tip design, which provides more efficient processing of narrower canals, lower possibility of transport, and blockage of canals, i.e., lower need for mandatory initial passability. The ONE Endo file is used for the initial extension and the EXO Endo for the final canal formatting<sup>1</sup>.

The Two Shape system (MicroMega, France) is made of T-wire by enhancing One Shape austenitic files but with a new asymmetric design that improves cutting efficiency and detritus evacuation. Uslu et al.<sup>37</sup> have found that T-wire technology is responsible for increasing cyclic fatigue resistance by 40% compared to One Shape files.

### C-wire

Heat-treated NiTi alloy is the basis for making the One Curve system (Micro-Mega, Besançon, France) with a single file in full rotation, which was created by different heat treatment processes of C-wire. The file has a CME, increased flexibility, and greater resistance to cyclic fatigue<sup>43</sup>. The

One Shape system (Micro-Mega, Besancon, France) showed 2.4 times higher resistance to cyclic fatigue compared to the previous generation<sup>43</sup>. The cross-section varies along the blade of the file, which improves the centering in the apical third of the canal and provides more efficient debridement<sup>43,44</sup>.

### Specific heat treatments of finished files

After making endodontic files, heat treatments can correct the defects that have occurred during the processing of the wire and affect the modification of the crystal structure of the alloy. Thermocycling of NiTi alloy allows martensitic transformation in two phases instead of one<sup>23</sup>. Furthermore, the phase transformation of austenite into martensite takes place first, after additional heat treatment due to the formation of the R-phase and deposition of Ti<sub>3</sub>Ni<sub>4</sub> particles; additional cooling of the alloy is necessary to form the martensite phase<sup>1,23</sup>.

### Blue-wire

Dentsply Tulsa Dental (Tulsa, OK, USA) introduced ProFile Vortex Blue in 2011, the first endodontic file with a recognizable blue color<sup>25</sup>.

Vortex Blue is a rotating system made of M-wire with increased fatigue resistance, better cutting efficiency, flexibility, and better centering along the canal<sup>14,20</sup>. The files were created by a new, protected wire treatment, which involves a two-stage transformation of the alloy at the austenitic phase temperature (38 °C). The recognizable blue color originates from the surface layer of Ti oxide (thickness 60 nm–80 nm), which, in addition to better cutting efficiency, also affects the increased resistance to wear<sup>14,20</sup>. Goo et al.<sup>45</sup> tested the influence of alloy type (stainless steel, conventional SE NiTi, M-wire, and Vortex Blue) on cyclic file fatigue in an artificially formed stainless steel canal and confirmed the best characteristics of Vortex Blue sets in terms of flexibility and resistance to fracture. The protected production process of Vortex Blue files reduces the SME compared to standard NiTi files but allows the default shape to be maintained during canal instrumentation<sup>45</sup>.

The Sequence Rotary and X1 Blue File systems (MK Life, Porto Alegre, RS, Brazil) were created in 2017. The systems include files created by multiple heat treatments of CM-wire (alternating heating and cooling), where a bluish hue that originates from a thin Ti oxide layer (60 nm–80 nm) is formed<sup>1</sup>.

The new generation of Reciproc Blue (VDW, Munich, Germany) files was created in 2016. This system combines the simplicity of the original RECIPROC concept of a single file with the application of reciprocal movements that result in greater safety in primary or re-endodontic treatment. Innovative heat treatment provides greater flexibility and easier and safer file progress in the canal<sup>10,46</sup>. The characteristic blue color, which leads to modifications of the molecular structure and an increase in flexibility and resistance to cyclic fatigue, is formed by heat treatment of the CM-wire<sup>10,46,47</sup>.

### Gold-wire

The unique heat treatment before and after file creation led to the development of the WaveOne Gold (Dentsply Sirona) system in 2011. In this process, the conventional alloy is first subjected to a heat treatment process (410 °C–440 °C, under a constant load of 3 kg–15 kg), and the finished file is then subjected to another thermal process (120 °C–260 °C). This technological process results in a surface gold color of the file (Ti oxide layer, 100 nm–140 nm), which significantly improves the flexibility and file resistance<sup>20</sup>.

The ProTaper Gold (Dentsply Sirona) system was created by subsequent heat treatment of files at a temperature of 370 °C–510 °C in 2013<sup>1</sup>. ProTaper Gold is similar to the ProTaper Universal system (size, convex triangular cross-section, and a progressive cone), but features a gold color which affects increased flexibility and resistance to cyclic fatigue, and ensures better file centering<sup>1,14,48</sup>. The golden color of the files originates from the layer of Ti oxide (thickness of 100 nm–140 nm); it affects the weaker effect of SM, so slight deformations are often noticeable in new, unpacked files<sup>20</sup>. This property allows files to retain the bent shape even after removal from the bent canal after processing<sup>14,20</sup>.

AF Blue (Fanta Dental CO., Ltd, Shanghai, China), the new NiTi file system, is made of specially heat-treated AF-wire (AFTM-H) with excellent mechanical properties (strength), flawless surface finish, higher resistance to cyclic fatigue, and good cutting efficiency. The extreme flexibility of these files prevents the occurrence of transport, and the variable cross-section ensures minimal radial contact and reduces the possibility of screwing<sup>44</sup>.

S-One (Fanta Dental CO., Ltd., Shanghai, China) is a heat-treated file used in the technique of single file in full rotation. The file has an unusual design with a tip diameter of #25 size and a 6% of a constant cone, i.e., an S-shaped cross-section<sup>24,26</sup>.

### Treatment by electric discharge machining of NiTi alloy

Electric discharge machining (EDM), or EDM technology, is a non-contact thermal erosion process used in the production of electrically conductive materials and involves controlled electrical discharge in the presence of insulating fluid.

Coltene introduced the Hyflex EDM system (Coltene/Whaledent, Cuyahoga Falls, OH) in 2016, which is made of NiTi CM 495 alloy and produced by spark technology<sup>37</sup>. The surface of the metal (NiTi alloy) is “melted” by this process, with partial evaporation leaving an eroded surface. The file is exposed to heat before or after ultrasonic cleaning (between 300 °C and 600 °C for 10 min to 5 hrs), which improves cyclic fatigue resistance by more than 700% at room or body temperature. Analysis of the surface structure of Hyflex CM and Hyflex EDM files, before and after processing of strongly bent canals, indicated fewer surface defects in Hyflex EDM systems<sup>37</sup>. These systems have different cross sections on the working part and provide greater

cutting efficiency, facilitate penetration, and reduce the risk of fracture<sup>20,37</sup>.

Sterilization allows these files to be recovered and restored to their original form. However, smaller files are usually permanently deformed, so special care should be taken when reusing them<sup>37</sup>.

Neoniti (Neolix, Chatres-La-Forêt, France), a new NiTi system with a single file in full rotation, is made of a special alloy subjected to subsequent heat treatment. The rectangular cross-sectional design and specific heat treatment significantly increase the flexibility and effect of SM. Treatment of the working part of the file by the EDM creates a rough surface and enhances the abrasive properties, activating the cutting efficiency<sup>44</sup>.

### Specific surface treatments after making files

Improvement in the physical and mechanical properties of files can be achieved not only by heat treatments after machining but also by specific surface treatments of finished files (ionic implementation, nitriding, cryogenic treatment, electropolishing)<sup>1,14</sup>.

Ion implantation of endodontic instruments in plasma was introduced in the late 1980s<sup>20</sup>. A highly negative pulsating voltage is applied to the plasma-submerged file in the vacuum chamber. In this way, ions are extracted from the plasma and penetrated on the surface of the file. Studies have shown the success of this procedure by incorporating argon, boron, and nitrogen<sup>1,14,20</sup>. Ionic implantation improves surface characteristics without affecting the superelastic properties of the alloy. Gavini et al.<sup>1</sup> have shown that ionic nitrogen implantation improves resistance to cyclic fatigue and that this treatment also leads to increased cutting efficiency, i.e., improved wear resistance.

Due to the higher affinity of Ti for binding to oxygen, prolonged exposure of the alloy to moderate temperature leads to the formation of a surface oxide film of TiO<sub>2</sub>, which is formed slowly<sup>20</sup>. The surface porous oxide film, which increases the stability of the surface layers of the alloy and protects against corrosion, is formed by coating endodontic files with a flexible protective layer of TiO<sub>2</sub> using the immersion sol-gel method<sup>14,49</sup>.

Titanium nitride, which consists of a thin outer layer of TiN and a thicker inner layer of Ti<sub>2</sub>Ni<sup>14,20</sup>, is formed by the method of thermal nitriding on the surface of files. Nitriding is performed at 250 °C (at 300 °C, the superelastic character of the file is lost), and the TiN layer on the files significantly increases the corrosion resistance in contact with 5.25% NaOCl<sup>14,25</sup>.

The deep dry cryogenic process increases resistance to corrosion and wear and improves the strength and microhardness of treated metals<sup>20</sup>. This method involves suspending the metal through a super-cooled bath with liquid nitrogen (-196 °C) and then gradually warming it to room temperature. The mechanism of action is twofold because the transformation of martensite into austenite is more complete after cryogenic treatment, and the deposition of smaller carbide particles within the crystal structure has also been observed<sup>14,20</sup>. Cryogenic file treatments affect significantly

higher microhardness and higher cutting efficiency without affecting wear resistance, with a significant improvement in the cyclic fatigue resistance of NiTi rotary files<sup>14</sup>.

Electropolishing is an electrochemical process for surface finishing of NiTi files and was first applied by FKG (La Chaux-de-Fonds, Switzerland) in 1999<sup>1,20</sup>. This procedure leads to the removal of surface irregularities (by dissolving metal ions) by immersing the files in an electrolyte bath (concentrated solution of high-viscosity sulfuric or phosphoric acid) at a controlled temperature. The passage of current causes the oxidation of the metal and its dissolution in the electrolyte, and a hydrogen release reaction takes place at the cathode. The formation of a thin passive layer and the dissolution of the surface leads to the removal of surface irregularities, which increases the resistance to cyclic fatigue and torsional loading of files and provides greater resistance to corrosion<sup>1,17,24,26</sup>. Examples of these files are RaCe systems (FKG, La Chaux-de-Fonds, Switzerland), available in several variations and clinical sets (RaCe, IRaCe, BioRaCe, Series ISO 10, Scout RaCe, BT RaCe). Such a design provides better cutting, reduces the possibility of screwing inside the canal, and provides better apical penetration of files<sup>1,17</sup>.

EndoSequence files (Brasseler, Savannah, GA, USA), created in 2009, were also subjected to electrochemical polishing and immersed in an ionic solution through which an electric current passes in order to remove all surface irregularities that occurred during the production process.

The results indicated greater resistance to corrosive defects in files with an electropolished surface and less prevalence of production defects on the work surface<sup>17,22,34</sup>. However, some authors state that file design has a more significant effect on file resistance to cyclic fatigue than the finishing itself<sup>1,49</sup>.

### Conclusion

Owing to the thermomechanical treatments of the alloy, the latest technological advances have enabled the development of new NiTi instruments (M-wire, files with CM, CM-wire, R-phase wires, Max-, T-, and C-wires), which with different start kinetics and the design features of the working part show improved flexibility and greater resistance to cyclic fatigue compared to traditional superelastic NiTi systems.

Conventional NiTi alloy usually contains an austenitic phase, and thermomechanically treated alloy contains different amounts of R-phase and martensite. Thermally modified alloy enables the production of more flexible rotary files with greater resistance to defects and fractures.

Improvement in the physical and mechanical properties of NiTi rotary files can be achieved by heat treatments after machine file making (Blue-wire, Gold-wire, EDM) or by specific surface treatment of files (ionic implementation, electropolishing, nitriding, cryogenic treatment).

Files with a dominant austenitic phase are superelastic and more suitable for processing straight or slightly bent canals, and such an alloy compensates for the reduced torsional resistance of the files to form the initial patency. Martensitic form files are more flexible and are characterized by in-

creased resistance to cyclic fatigue and lower torque, and are suitable for processing bent and complex canal systems.

Files made of heat-treated CM-wire show greater flexibility and a weaker SME. Therefore, they can be bent before application into the canal and thus more efficiently clean and shape bent canals.

The dental practitioner is the most important factor for the success of the endodontic intervention. In addition to essential skills, they must know the complex anatomy of the canal and the characteristics of the file design, choose the right set of files and adapt the instrumentation technique to each individual case.

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