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Recent advances in metallurgy and design of rotary endodontic instruments: a review

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ABSTRACT

A variety of instruments are available for the extirpation of the pulp and the instrumentation and preparation of the root canal. Recently, nickel-titanium (NiTi) alloy is utilised for the manufacturing of endodontic instruments. Compared to other metals, these alloys are highly flexible, which significantly enhances ease of canal shaping. This review article gives an account in the advances of NiTi endodontic instruments with an emphasis on metallurgical, mechanical properties, the design features of each generation with a special focus on the latest generations of NiTi instruments.

1. Introduction

To overcome some of the undesirable characteristics of stainless-steel files, a new generation of endodontic instruments made from nickel-titanium has added a new dimension to the practice of endodontics. The NiTi alloys started at the beginning of the '60s by W. H. Buehler, a metallurgist investigating nonmagnetic, salt resistant and waterproof alloys for the space program at the "Naval Ordinance Laboratory", in Silver Springs, Maryland, USA. This was named "NITINOL based the elements from which the alloy was composed; Ni for Nickel, Ti for titanium and NOL for the Naval Ordinance Laboratory. Nitinol is the names given to a family of intermetallic alloys of Ni and Ti, which possesses unique properties such as shape memory and superelasticity [1]. The first investigation of nickel-titanium in endodontics was reported in 1988 by Walia, Brantley and Gerstein using #15 files fabricated from nickel-titanium orthodontic alloy.

The unique property of super-elasticity allows to carry out extraordinary conservative shapes, and it can also be better centered. Furthermore, it provides less canal transportation and in this manner with more regard of the original anatomy. Advanced instrument designs have been developed to reduce the preparation time, improve working safety, and create a conical flare of preparations and continuously tapered shapes. The advanced instrument designs include with non-cutting tips, radial lands, different cross-sections, superior resistance to torsional fracture and with varying tapers [2].

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2. Temperature Induced Phase Transformation

The crystal structure of NiTi alloy at high-temperature range (10000C) is stable with body centered cubic lattice (BCC), which is referred to as the austenite phase or parent phase. Nitinol shows dramatic changes in its yield strength, modulus of elasticity (MOE), and elastic resistivity on cooling through a critical transformation temperature range (TTR). Also, changes the crystalline structure occurs on reducing the temperature through this range. This change in crystalline structure is known as martensitic transformation. This phenomenon causes a change in the physical properties of the alloy and gives rise to shape memory characteristic (Figure 3) [3].

The atoms in this file are rearranged into a closely packed hexagonal array on placing into a curved canal. This atomic rearrangement imparts a more flexible character with a martensite crystal structure to the alloy. The molecular transition enables these files to bend easily around severe curves without permanent deformation. The alloy reverts to its original austenite form on the removal of the stress, and this is called as stress-induced martensitic transformation, which is a unique property of NiTi alloy and thus makes this material suitable for use in rotary endodontic instruments [4].

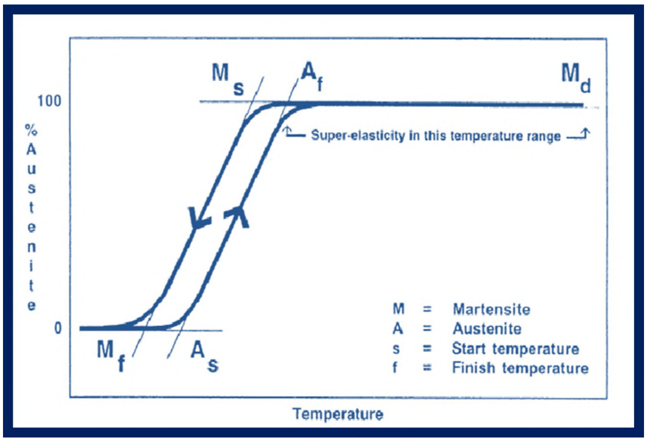

Figure 1. Temperature dependent transitions from austenite to martensite; where A_s: The starting temperature for transformation to austenite, A_f: The temperature at which transformation to austenite is finished. M_s: The starting temperature for transformation to martensite. M_f: The temperature at which transformation to martensite is finished.

3. Strategies in the alteration of NiTi alloy

In recent years, new forms of NiTi are developed by modifying the alloy by variations in metal processing and file manufacturing or correcting the surface defects.

3.1 Plasma immersion ion implantation

Conrad *et al.* and Tendys *et al.* introduced Plasma immersion ion implantation (PIII) in the late 1980s. The specimen is introduced in a chamber and immersed in the plasma, and a highly negative pulsating voltage is then applied to the sample. Briefly, it is a line-of-sight process in which ions are extracted from plasma, accelerated, and bombarded into a device. Ionic implantation brought differences in surface characteristics, an increase in the cutting efficiency, and improved wear resistance as shown by Rapisarda *et al.* [5].

3.2 Oxide formation on NiTi/Titanium oxide coating

Titanium has a higher affinity with oxygen compared to Nickel. So, with increased exposure time at moderate temperature, the oxide formation is composed mainly of TiO_2 with slow formation and growth. A study on the mechanical behaviour of the endodontic instruments and its corrosion resistance in Sodium Hypochlorite (NaOCl) solution was carried out by Aun DP *et al.* [6]. They observed an improvement in cutting efficiency and high resistance to corrosion on immersing in NaOCl solution. The coated instruments showed better performance in fatigue life after corrosion, so they concluded that this characteristic should be maintained since the TiO_2 layer can support relatively large deformations. Coating the endodontic instruments with a flexible TiO_2 protective layer with the help of dip-coating sol-gel method improves cutting efficiency, corrosion behaviour and resistance to fatigue failure.

3.3 Thermal nitridation

Powder Immersion Reaction Assisted Coating (PIRAC) is a nitriding method producing TiN on NiTi. Such modified surface consists of an outer layer of TiN which is thin and a thicker Ti2Ni layer underneath [7]. By placing a TiN layer on commercial rotary NiTi instruments corrosion resistance of files placed in contact with 5.25% NaOCl significantly increases.

3.4 Cryogenic treatment

Various metals are treated with deep dry cryogenic methods to enhance resistance to corrosion and wear.

These methods also improve strength and microhardness of metals. The entire cross-section of the instrument is affected rather than no change in the elemental crystalline composition on the surface of the alloy [8]. There are two mechanisms involved. In the first mechanism, the crystalline transformation from austenitic to complete martensitic following CT occurs. In the second mechanism, finer carbide particles precipitate within the crystalline structure (Figure 2). Kim *et al.* [9] observed that cryogenically treated instruments had significantly higher microhardness.

3.5 Electropolishing / Reverse plating

Electropolishing (EP) is a typical surface treatment process for final finish during the manufacturing of NiTi instruments. This process includes alteration of the surface chemistry and morphology takes place as surface imperfections are removed as dissolved metal ions. Once, the instrument is immersed in a temperature -controlled bath of electrolyte it serves as the anode when connected to the positive terminal of a direct current power supply, and the negative terminal is attached to the cathode. The surface of a metal oxidises as the current passes and dissolves in the electrolyte. A reduction reaction is observed at the cathode that normally produces hydrogen. Most often the electrolytes used are mixtures of concentrated solutions of sulfuric/phosphoric acid with a high viscosity. Bare NiTi surfaces are produced from Ti oxides with Ni concentrations from 2% to 7% depending on the electrolytes and regimes employed. In the process, the metal exhibits better corrosion resistance along with improved surface characteristics. Anderson *et al.* reported that the instruments with electropolishing show better resistance to cyclic fatigue loads and poor resistance to static torsional loading. The benefits of electropolishing are likely caused by a reduction in surface irregularities that serve as points for stress concentration and crack initiation [10]. Lopes *et al.* found significant increases in cyclic fatigue resistance and exhibiting fine surface cracks which assumed an irregular or zigzag path, that EP instruments demonstrated. In contrast, the non-EP files had cracks running along the machining grooves [11].

4. Modifications in the microstructure of alloy by thermo-mechanical treatment

Thermo-mechanical treatment, is a metallurgical process, which involves in combining both the mechanical or plastic deformation process (compression or forging, rolling etc.,) and the thermal processes (heat-treatment, water quenching, heating and cooling at various rates) into a single process.

4.1 Thermal processing during the manufacturing of alloy

The mechanical behaviour of NiTi alloy is determined by the relative proportions and characteristics of the microstructural phases. Heat-treatment (thermal processing) is a fundamental approach towards regulating the transition temperatures of NiTi alloys and affecting the fatigue resistance of NiTi endodontic files. More the

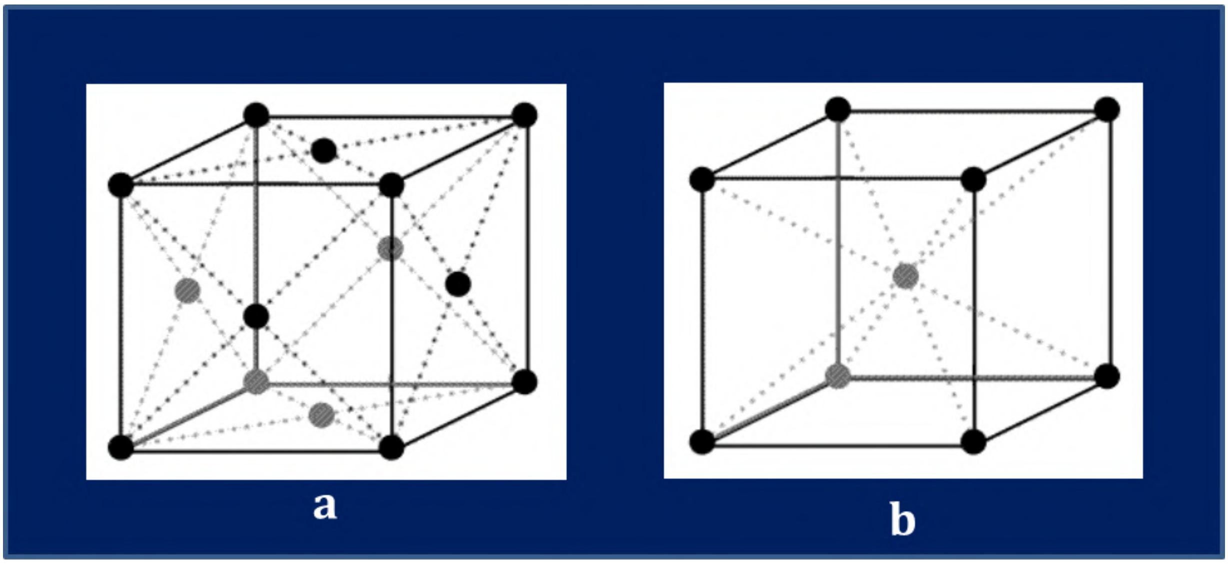

Figure 2. Crystalline structure changes from the cryogenic treatment. Where; a. Before treatment: Face-centered cubic austenitic structure, and b. After treatment - Cell-centered structure

martensitic NiTi alloy more is the flexibility and fatigue resistance of an instrument as it produces a better arrangement of the crystal structure and changes in the relative percentage of phases present in the alloy as found by De Vasconcelos [12]. Development of the next-generation endodontic instruments is made on the enhancements in these areas of material management.

4.2 M-Wire

M-Wire was developed to produce superelastic NiTi wire blanks that contain substantial stable martensite under clinical conditions M-Wire was developed. Martensite, stress-induced martensite (SE), and austenite are three different forms of NiTi. Softness, ductility and easy deformation are observed when the material is in its martensite form. The austenitic NiTi is strong and hard, whereas SE NiTi is highly elastic. The martensitic phase transformation has excellent fatigue resistance because of the energy absorption characteristics of its twinned phase structure. In 2007, M-wire (Dentsply Tulsa- Dental Specialties, Tulsa, OK, USA) introduced and it contains portions that are in both the deformed and micro twinned martensitic, pre-martensitic R-phase, and austenite while maintaining a pseudoelastic state [13]. The austenite-finish temperature (A_f) of M-Wire is in the range of $45^0C–50^0C$ [14]. This temperature range indicates that the instruments manufactured from M-Wire would be necessary for the martensitic phase at room temperature. Various M-Wire instruments include Dentsply's ProFile GT Series X, ProFile Vortex, and ProTaper Next files, Path Files, WaveOne and Reciproc (VDW, Munich, Germany). Gao *et al.* [15] studied the effect of cyclic fatigue loading on M-wire and a regular SE wire at two different rational speeds. They found multiple crack-initiation sites with more than 50% of broken files, which are made from SE wire and a single crack initiation on files made of M -Wire.

4.3 R-Phase

R phase is formed during the forward transformation of martensite to austenite on heating and reverse transformation from austenite to martensite on cooling. Upon heating, martensite will start transforming to R-phase at R_s temperature, and this transformation will be finished at the R_f temperature. On further heating, R-phase starts transforming to austenite at the A_s temperature, and transformation is finished at A_f temperature. If heated above A_f temperature, it will be converted entirely to austenite. Then, upon cooling to a sufficiently lower temperature, the alloy starts transformation from austenite to R-phase at the R_s temperature and this transformation will be finished at the R_f temperature. By further cooling, the R-phase starts transforming to martensite at Ms temperature and finished at M_f (Figure 3) [16]. The alloy obtains greater strength and a lower modulus of elasticity on comparison with stainless steel. Therefore, instruments made with R-phase wire are more flexible than stainless steel [17]. Twisting of the wire can be performed once the R-phase is identified as it optimises the grain structure in the metal. The grinding process weakens the metal's structure at the molecular level that results in creating microfractures on its surface, leading to fracture of files [18]. In 2008, SybronEndo (Orange, CA, USA) developed Twisted Files (TF) and K3XF files by twisting the intermediate alloy followed by a heat-treatment process. R-phase exhibits lower shear modulus compared to martensite and austenite phases. Further, the transformation strain is also less than one-tenth to that of martensitic transformation. Instruments made with R phase are fully austenitic at ambient and body temperatures [19] and also imparts greater flexibility and increased resistance to flexural fatigue as stated by recent reports [20]. On the contrary, Park *et al.* [21] observed that this manufacturing method fails in providing any beneficial effect with regard to torsional fracture.

4.4 Controlled memory NiTi alloys (CM Wire)

CM Wire (DS Dental, Johnson City, TN) was introduced in 2010. It is a novel NiTi alloy with flexible properties. These CM wires are manufactured by a proprietary thermo-mechanical process, which allows the instruments to be pre-curved before they are placed into the root canals. In addition, this process also increases the flexibility, the transformation temperatures (A_f to about 50°C), reduces the shape memory, and also helps in obtaining stable martensite at the body temperature. However, they revert to their original shape on sterilization. Various CM NiTi file systems include Hyflex CM (ColteneWhaledent, USA), Typhoon CM (Clinician's Choice Dental Products, USA) and ProFile Vortex Blue (Dentsply).

During clinical use, the conventional NiTi files are in the austenite phase as their A_f temperature is at or below room temperature. However, the A_f of CM files is certainly above the body temperature, which results in the formation of both martensite and R-Phases in addition to the austenite phase. The Hyflex CM and Typhoon CM instruments contain a combination of martensitic R-phase

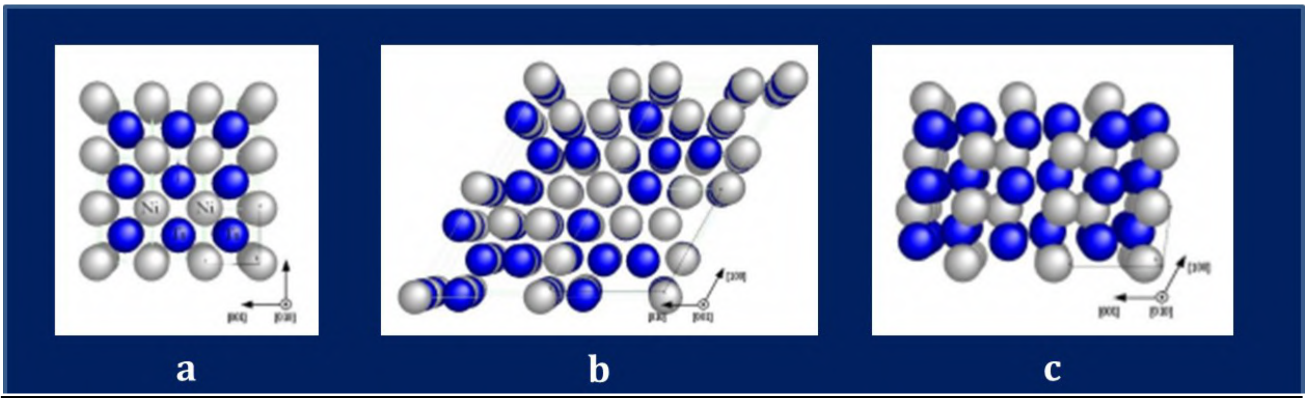

Figure 3. Experimental structures of NiTi alloys. a. B2 austenite PM3-M, b. R-phase P3, and c. B19 martensite p21/m (cubic coordination); Where grey and blue coloured spheres are Ni and Ti atoms respectively.

and austenitic phase at body temperature as their A_f temperatures are slightly above the body temperature [22]. Shen *et al.* [23] reported that instruments made from CM wires exhibited around 300% to 800% more resistance to fatigue failure compared to instruments made from conventional NiTi wires. Longer fatigue life observed with the square configuration of NiTi instruments made from CM Wire than the triangular configuration.

4.5 Thermal processing after machining of files / Post-machining heat-treatment

Thermal processing used to overcome the defects occur during the machining process and also to modify the crystalline structure of alloys. Thermocycling of NiTi alloys causes the martensitic transformation to occur in two stages instead of in a single stage. The stage-1 transformation (A-M) takes place in Ni-rich NiTi alloys. Stage-2 transformation (A-R-M) takes place after the additional heat-treatment, which precipitates finely dispersed Ti_3Ni_4 particles in the austenitic matrix. Accordingly, the R-phase is formed instead of martensite due to the presence of fine dispersed Ti3Ni4 particles. Therefore, it necessitates additional cooling of alloy to form martensite and hence, martensitic transformation occurs in 2 steps (A-R-M) [24].

4.6 Vortex blue

Vortex blue is a newly developed NiTi rotary instrument with improved fatigue resistance, cutting efficiency, flexibility, and canal centering capability, and that is made from M-Wire [25]. Its A_f temperature is around 38°C. Vortex blue has a 2-stage transformation, as observed in studies. Unique "blue colour" is seen by Vortex Blue instruments on comparison with traditional SE NiTi instruments. Proprietary manufacturing process leads to the "blue-colour" oxide surface layer of Vortex Blue files. Compensation for the loss of hardness along with improvement in the cutting efficiency and wear resistance is achieved by the relatively hard titanium oxide surface layer on the Vortex Blue instrument.

4.7 ProTaper Gold (PTG)

The geometries of both ProTaper Gold (PTG) ProTaper Universal (PTU) are the same with a convex triangular cross-section and progressive taper. The files are heat-treated after their manufacturing at around 370-510°C for a variable period. Similar to CM wire, these files exhibit two stage specific transformation behaviour and high A_f temperature about 50°C [26] that offers increased resistance to cyclic fatigue loads for PTG instruments compared to instruments made with PTU. Less shape memory is observed with PTG than NiTi so unopened package of files exhibiting a slight degree of curvature is not a surprising feature. It is an advantage rather than a defect as supposed by the manufacturer. The file follows the root canal anatomy being shaped upon removal from a curved canal.

4.8 WaveOne Gold

Unique heat-treatment before and after file manufacturing led to the development of WaveOne Gold. The SE NiTi alloy is subjected to a unique heat treatment process in a temperature range of about 410° to 440°C, under constant strain (3-15 kg). The finished instrument is subjected to a second heat treatment process in a range of 120°C to 260°C, after machining the working portion of the file. The A_f temperature of WaveOne Gold is in the range of 40°C-60°C.

Numerous manufacturers suggested that this technology improves the flexibility and strength of the instrument. Torsional resistance could be enhanced due to off-centred parallelogram-shaped cross-section design.

4.9 Hyflex EDM

Coltene Whaledent manufactured Hyflex ED based on the EDM method. EDM stands for electrical discharge machining (EDM), which is a noncontact thermal erosion process, that is used to machine electrically conductive materials with the help of controlled electrical discharges. This process generates the electrical sparks, which results in a local melting and partial evaporation of small portions of material resulting in the formation of a typical crater-like surface finish. Then, the instrument is cleaned through ultrasonic in an acid bath, followed by a heat treatment process at a temperature ranging between 300-600°C for 10 min to 5 hours before or after the cleaning process. A_f temperatures over 52°C are observed with EDM files [27]. Early material failure is avoided by producing a non-directional surface finish by EDM process.

On comparison with other rotary NiTi instruments, HyFlex may be up to 300% more fatigue- resistant as reported by manufacturer. The instrument regains its shape by sterilization. Peters *et al.* [28] reported that more than half of the plastically deformed instruments recovered to their original shape during sterilization. However, sterilization had shown no effect on the small instruments in retaining their shape. Therefore, care should be taken regarding reuse of small HyFlex rotary instruments. Less apical pressure against canal walls is required to be applied than with conventional SE NiTi files of the same size and taper.

4.10 K3 XF

In 2011, SybronEndo developed K3XF by taking advantage of R-phase technology. Fabrication takes place by a grinding process rather than a twisting process. A Special heat treatment process is performed on K3XF files after the grinding process to enhance flexibility and strength. This heat-treatment also modifies the crystalline structure of the alloy to accommodate some of the internal stress caused by the grinding process [29]. K3XF instruments undergoing post-machining heat treatment differs them from K3, but both are identical in shape. Since K3XF instruments have an A_f temperature below 37°C therefore, it has an austenite structure at body temperature and exhibits superelastic property during clinical application. The heat treatment processing used for K3XF modifies the transformation temperature by releasing crystal lattice defects and diminishing internal strain energy. K3XF instruments have numerous micropores with various diameters on the surface of the instrument flute. These small pores serve as a local stress/strain discontinuity from which the crack nucleates and does not contribute to the failure [30].

4.11 XP Endo Shaper

FKG Dentaire, La-Chaux-de-Fonds, Switzerland introduced XP endo shaper in 2016 with 0.30 diameter and 0.1 taper that could expand to 0.4 taper. At room temperature, these instruments are relatively straight in their M-phase (martensitic state) and change to a curved shape on exposed to intracanal temperature due to a phase transformation to A-phase (austenitic state). The martensite to the austenite phase occurs naturally in the body temperature (32°C and 37°C) with Af temperature at 35°C. In a dynamic state, the instrument has a twisted shape, with several twists twisted along its length. Shape memory effect is exhibited when these instruments are inserted into the root canal (M-phase to A-phase) and possess superelasticity during preparation. The curved shape enables the preparation of complex root canal morphologies with the potential to adapt to canal irregularities.

5. Conclusion

Thermomechanical treatment of NiTi alloy allows a transition in the phase composition leading to the appearance of martensite or R-phase under clinical conditions. While M-Wire and R-phase instruments maintain an austenitic state, CM Wire, as well as the Gold and Blue heat-treated instruments are composed of substantial amounts of martensite. High torque values at fracture are revealed by austenitic instruments possessing superelastic properties. Thus, to shape straight or slightly curved root canals, these files are appropriate. Use of austenitic alloy in pathfinding instruments compensates for the decreased torque resistance caused by the smaller diameter of these files. Martensitic instruments are more flexible with enhanced resistance to cyclic fatigue, and they also reveal a greater angle of rotation but lower torque at fracture due to an increased amount of the martensite phase. In complex curvatures and root canal anatomies, cyclic fatigue is known to occur more likely. Thus, in cases of severely curved root canals or those with double curvature martensitic instruments should be preferred. Moreover, when trying to bypass ledges, Martensitic instruments are useful.

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