



## Recent advances of metallurgy in endodontics: 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 which emphasis on metallurgical, mechanical properties, the design features of each generation with a special focus on the latest generations of nickel titanium(NiTi) instruments.

### INTRODUCTION

To overcome some of the undesirable characteristics of stainless-steel files, a new generation of endodontic instruments made from nickel-titanium which has added a new dimension to the practice of endodontics. The NiTi alloys started at the beginning of the 60's by W. H. Buehler, a metallurgist investigating non-magnetic, salt resistant and water proof alloys for the space program at the "Naval Ordnance Laboratory", in Silver Springs, Maryland, USA. This was named "NITINOL based elements from which the alloy was composed; Ni for Nickel, Ti for titanium and NOL for the "Naval Ordnance 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 super elasticity [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]

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[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]

## **MATERIALS AND METHODS**

A comprehensive electronic search was conducted in the PubMed database. All peer-reviewed and full length studies in the English language were included. The search was performed using following keywords: “nickel-titanium in Endodontics” or “rotary in Endodontics”. Articles included were indexed in Google Scholar, PubMed Central, Scopus and/or Web of Science database.

## **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.

## **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].

## **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<sub>2</sub> 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<sub>2</sub> layer can support relatively large deformations. Coating the endodontic instruments with a flexible TiO<sub>2</sub> protective layer with the help of dip-coating sol-gel method improves cutting efficiency, corrosion behaviour and resistance to fatigue failure.

## **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 Ti<sub>2</sub>Ni 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.

## **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 crystal-line structure. Kim et al.[9] observed that cryogenically treated instruments had significantly higher microhardness.

### **ELECTROPOLISHING / REVERSE PLATING ELECTROPOLISHING (EP)**

It 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 zig-zag path, that EP instruments demonstrated. In contrast, the non-EP files had cracks running along the machining grooves [11].

### **MODIFICATIONS IN THE MICROSTRUCTURE**

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.

### **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 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.

### **M-WIRE**

M-Wire was developed to produce super elastic 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 pseudo elastic state [13]. The austenite-finish temperature ( $A_f$ ) of M-Wire is in the range of 450°C–500°C [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 rotational 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

### **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  $50^\circ\text{C}$ ), 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 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.

Thermal processing after machining of files/ Post machining heat-treatment- It is used to overcome the defects occur during the machining process and also to modify the crystalline structure of alloy. Thermocycling of NiTi alloys causes the martensitic transformation to occur in two stages instead of 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  $\text{Ti}_3\text{Ni}_4$  particles in the austenitic matrix. Accordingly, the R-phase is formed instead of martensite due to the presence of fine dispersed  $\text{Ti}_3\text{Ni}_4$  particles. Therefore, it necessitates additional cooling of alloy to form martensite and hence, martensitic transformation occurs in 2 steps (A-R-M) [24]

### **NEWER NITI ROTARY SYSTEMS**

The examples of the some newer NiTi rotary systems manufactured using various modifications in metallurgy are as follows:

**Vortex blue** It is 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  $38^\circ\text{C}$ .

**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  $370\text{-}510^\circ\text{C}$  for a variable period. Similar to CM wire, these files exhibit two stage specific transformation behaviour and high  $A_f$  temperature about  $50^\circ\text{C}$  [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.

**Hyflex EDM-ColteneWhaledent** 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. Af 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.

**10 K3 XF** - In 2011, Sybron Endo 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 Af temperature below 37°C therefore, it has an austenite structure at body temperature and exhibits super elastic 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].

**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 super elasticity during preparation. The curved shape enables the preparation of complex root canal morphologies with the potential to adapt to canal irregularities.

## **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 to shape straight or slightly curved root canals, these files are appropriate. Martensitic instruments are more

flexible with enhanced resistance to cyclic fatigue. 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, and also to bypass ledge martensitic instruments should be preferred.

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