ISSN: 0975-833X

Available online at http://www.journalcra.com

International Journal of Current Research
Vol. 9, Issue, 05, pp.51300-51306, May, 2017

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

RESEARCH ARTICLE

REVIEW AND CLASSIFICATION OF HEAT TREATMENT PROCEDURES AND THEIR IMPACT ON MECHANICAL BEHAVIOR OF ENDODONTIC FILES

***Dr. Christia M. Aoun, Pr. Walid B. Nehme, Pr. Alfred S. Naaman and Pr. Issam T. Khalil**

Department of Endodontics, Faculty of Dentistry, Saint Joseph University, Beirut, Lebanon

ARTICLE INFO

Article History:
Received 25th February, 2017
Received in revised form
04th March, 2017
Accepted 10th April, 2017
Published online 31st May, 2017

Key words:
Endodontics (MeSH),
Heat treatment;
Metallurgy (MeSH),
Nickel Titanium,
Technology (MeSH).

ABSTRACT

Objectives: The article presents an updated literature review on NiTi endodontic instruments in order to explain and analyze the influence of heat treatment process on mechanical properties of Niti shape memory alloys. The reader should understand the clinical implications of thermal heating of endodontic intruments such as enhanced flexibility and safety.

Methods: The literature concerning the influence of heat treatment on mechanical properties of NiTi alloy has been analyzed using selected criteria. A closer look at multiple patents was important to understand the effects of heating on crystalline microstructure and phase transformation properties.

Results: The main advantage gained by heat treatment process is the increase of Af (Austenite finish) temperature of the alloy. If "Af" is superior to body temperature, the file will be in a mixed martensitic, R-phase and austenitic structure in intracanal temperature. Therefore, heat treated instruments showed a significant increase in flexibility and flexural fatigue resistance. Only few reports describe the mechanical behavior under torsional loads. Interestingly, modifications in crystalline structure seem to have an influence on angle of deflection and torque load, which are two determinant parameters of torsional fracture.

Conclusion: An analysis of the different effects of heat treatment on Niti instruments is presented, thus enabling an easier understanding of mechanical properties of new thermomecanically treated alloys.

Clinical relevance: The theme is relevant for clinical Endodontics since the variety of instruments is great and clinicians need a better understanding of the different ways of their manufacturing for a better use during endodontic treatment.

Citation: Dr. Christia M. Aoun, Pr. Walid B. Nehme, Pr. Alfred S. Naaman and Pr. Issam T. Khalil, 2017. "Review and classification of heat treatment procedures and their impact on mechanical behavior of endodontic files", *International Journal of Current Research*, 9, (05), 51300-51306.

INTRODUCTION

Nitinol wire instruments were introduced to the field of endodontics because their increased flexibility and bending properties over traditional stainless-steel instruments (Walia, 1988). The mechanical behavior of NiTi alloy is determined by the relative proportions and characteristics of the microstructural phases (Shen, 2013). In recent years, several novel thermomechanical processing and proprietary manufacturing technologies have been developed to optimize the microstructure and the flexibility of NiTi alloys (Gutmann, 2012). Numerous investigations explored the mechanical behavior of the Niti shape memory alloys, but few addressed the influence of heat treatment. However, there is still lack in understanding the fundamental mechanisms related to the thermomechanical treatment. This focused review will explore different patented manufacturing process, address the impact on instrument mechanical properties, and classify the different file systems accordingly.

Literature Search strategy

A search of the existing literature was performed on PubMed, Cochrane Library and EBSCO electronic databases. The research included dental publications, materials science papers treating metallurgy, and engineering books, all written in English. An additional hand search was extensively performed in the Journal of Endodontics, International Endodontic Journal, American Journal of Orthodontics and Dentofacial Orthopedics, International Journal of Engineering Research and Development, Dental Materials (Official Publication of the Academy of Dental Materials), Materials Science and Engineering: A Journal, Metallurgical and Materials Transactions A Journal, The journal of " Reviews on advanced materials science", Acta/ Scripta Metallurgica et Materialia

---

****Corresponding author:*** **Dr. Christia M. Aoun,**
Department of Endodontics, Faculty of Dentistry, Saint Joseph University, Beirut, Lebanon

Journal, Metallurgy - Advances in Materials and Processes (Book). After the removal of duplicate articles, title review, and abstract selection, full-text articles were retrieved to verify that the topic was pertinent. Selected articles were reviewed by the authors, and inclusion criteria were that the articles should analyze any kind of endodontic instrument which has undergone heat treatment.

Heat treatment effects on crystalline microstructure and phase transformation properties

Nitinol alloys configure in different temperature dependent structures. At higher temperatures, Nitinol is in an austenitic state known to be that of a face centered cubic lattice. At lower temperatures, Nitinol is in a martensitic crystalline structure which is a monoclinic distorted structure. The distorted structure allows for the material to be deformed at greater angles and working conditions than that of the austenitic Nitinol in same conditions. The transformation between phases is initiated by heat and stress (Shen, 2013; Johnson, 2013; Thompson, 2000). Manufacturers have been using complex procedures to modify the alloy transformation temperatures such as Ms, Mf, As, Af, Rs, and Rf and consequently improve its mechanical performance (Miyai, 2006). According to transformation temperatures, microstructural composition at working environment and previous thermodynamical treatments a new classification is proposed

The first generation (SE files)

These files are in Austenic phase with Af temperature below body temperature and exhibits Super Elastic properties. Studies of the metallurgical structure of SE files with Conventional differential scanning calorimetric (DSC) analyses showed that the Af for most SE NiTi files is at or below room temperature (16-31 ˚C) (Brantley, 2002; Brantley, 2002; Zhou, 2012; Alapati, 2009; Shen, 2011 and Hou, 2011) and that these instruments are in the austenitic superelastic condition. Af of The ProFile (Dentsply Tulsa Dental, Tulsa, OK) and Lightspeed (Lightspeed Inc, San Antonio, TX, USA) instruments was near 25 ˚C (7). The Af temperatures for EndoSequence (Brasseler, Savannah, GA, USA), Profile, and Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT) files were respectively 31.13 ˚C, 16.98 ˚C and 16.22 ˚C (11). According to Alapati *et al*, Af of ProFile GT is 21 ˚C and of ProTaper Universal (PU) (Dentsply Tulsa Dental, Tulsa, OK) is 17 ˚C (10). Af of K3 (SybronEndo, Orange, CA, USA) (3.88 ± 3.21 °C) (Hou, 2011).

The second generation

In this group, several proprietary thermomechanical procedures have been developed to obtain SE wire blanks that contain the stable martensite phase under clinical conditions. Thermal processing, in the manufacturing of the alloy, produces a better arrangement of the crystal structure and alter the relative percentage of phases present in the alloy (Shen, 2013 and Shen, 2013). Heat treatment process generally leads to finely spread NiTi particles in the matrix (Otsuka, 2005) and shifts upward the Af of the alloy producing a different crystallographic percentage of martensite and or R-phase and or austenite near body temperature (Alapati, 2009). Depending on the thermodynamical treatment of the wires prior or during manufacturing, three different wires were produced.

M-Wire

Introduced in 2007 by Dentsply Tulsa Dental specialties, is reported to undergo a series of heat treatment and annealing cycles during the drawing of the wire (Kell, 2009). This cycling process aim to stabilize the crystalline structure of the Nitinol in its more martensitic condition at body temperature (Jordan, 2015). Different analytical methods confirmed that M-Wire contained austenite, martensite, and R-phase. The relative proportions depended on the processing conditions (Alapati, 2009). On scanning transmission electron microscopy martensite and perhaps R-phase were found in the cross-sections of M-Wire (Brantley, 2002). In a microstructural study, the classic lenticular appearance of martensite has been identified in the microstructure of M-Wire (Alapati, 2009). The austenite-finish temperature (AF) of M-Wire is around (45°C – 50°C) as shown by TMDSC analysis by Alapati *et al* (Alapati, 2009). The temperature range for phase transformation, suggests that these instruments made from M-Wire would be essentially in the martensitic phase at room temperature.M-Wire instruments include Dentsply's ProFile GT Series X (Af above 40°C) (18) ProFile Vortex (Af around 50 °C) (11), ProTaper Next files (PTN) (Af around 46.5 °C) (19) and PathFiles and WaveOne (WO) and Reciproc (VDW, Munich, Germany).

R phase

In 2008, SybronEndo (Orange, CA, USA) developed a new manufacturing process aiming to transform raw NiTi wire in the austenitic phase into an R-phase and to stabilize the R-phase at higher temperature. This proprietary twisting process with concurrent heat treatment imparts superior mechanical characteristics (Yum, 2011 and Kim, 2010). R-phase possesses lower shear modulus than martensite and austenite, and the transformation strain for R-phase transformation is less than one tenth that of martensitic transformation (Wu, 1990). At ambient and body temperatures, "R phase" instrument is fully austenite. The Af temperature of Twisted files TF is ranging between (17.62-18.88°C) (Hou, 2011). Moreover TF present a 2-step transformation through an apparent R-phase .The flexibility of the files could be related to the 2 phase transformation (Shen, 2011).

Controlled memory NiTi wire (CM Wire)

CM wire (DS Dental, Johnson City, TN) was introduced in 2010. A proprietary thermomechanical process aimed to increase the flexibility , reduce the shape memory ,raise the transformation temperatures (Af to about 50 °C) and obtain stable martensite at the body temperature (Santos, 2013). The chemical composition of CM wires and SE wires was studied. Zhou *et al.* (Zhou, 2012), stated after EDS results that the composition of CM and SE wires could be considered as the same. Testarelli *et al* found that CM has a lower percent in weight of nickel (52 Ni %wt) compared to the common 54.5–57 Ni % wt of the great majority of commercially available SE (Testarelli, 2011). Recent studies found that the Af temperature of Hyflex CM (Coltene Whaledent, Cuyahoga Falls, OH) and Typhoon CM (TYP; Clinician's Choice Dental Products, New Milford, CT) was about 47°C, and 55 °C respectively suggesting that this instrument at body temperature will be in a mixed martensitic R-phase and austenitic structure (de Vasconcelos, 2016; Shen, 2013). These data are consistent with the previous studies, which showed that instruments made from SE -NiTi exhibit an austenitic phase at room temperature, whereas MW and CM instruments, in addition to the austenite, also contained martensite and R-phase (Pereira, 2012).

The third generation: Post machining heat treatment

Recently, a new heating process after the machining of the files has been used to overcome machining process defects, and to modify the crystalline phase structure (Brantley, 2012). It has been reported that after thermal cycling, the martensitic transformation of NiTi alloys occurs in 2 stages (Alapati, 2009; Shen, 2011; Shen, 2013), instead of one. The 1-stage transformation (A-M) happens in Ni-rich NiTi alloys, whilst 2-stage transformation (A-R-M) happens after additional heat treatment. The heat treatment forms finely dispersed Ti3Ni4 precipitates in the austenitic matrix. Consequently, the R-phase is formed in preference to martensite due to the presence of Ti3Ni4 fine particles. However, the alloy needs additional cooling to form martensite, and hence, martensitic transformation occurs in 2 steps (A-R-M) (Otsuka, 2005). This process was implied to several systems by Dentsply Tulsa Dental Specialties such as:

Vortex Blue files are manufactured from 508 nitinol , then after machining, they undergo a special heating and cooling process discussed in the patent described by Gao (Gao, 2011), Its Af temperatures is around 38 °C (Shen, 2015), Studies (Berendt, 2007 and Shen, 2015), have shown that vortex blue had a 2-stage transformation. This can be understood by considering that R-phase is another potential martensite candidate that possesses a lower shear modulus and a shorter transformation strain.

TRUShape: is a novel heat-treated nickel- titanium file with a unique S-curve shape and a decreasing taper (Peters, 2015). The flutes are ground into blanks from commercially available nickel titanium then heat treatment is applied to shape set a file into characteristic bends. Temperature is at least about 300 ˚C for a time period of at least about 1 min to shape set the portion of the shaft thereby forming a shape set non linear file (Ammon, 2014). Af temperature of (31+/- 4) (de Vasconcelos, 2016). ProTaper Gold (PTG): Post heat treatment is applied after the flutes of a file have been manufactured. The temperature used is in a range of 370-510° C for a variable period of time (typically 10-60 min, depending on file size and taper) (Gao, 2011). Files exhibits 2-stage specific transformation behavior and high Af temperature around 50 ˚C (Hieawy, 2015). WaveOne Gold is the result of a unique heat-treatment prior and after file manufacturing. The super elastic Niti alloy is subjected to special heat treatment under constant strain in a range of 3-15 kg in a temperature range of about 410° to 440° C. After machining the working portion of the file, the finished instrument is heat treat a second time in a range of 120° C to 260°C. The Af temperature between 40°C - 60°C.(27) Other manufacturers are also applying this process:
Hyflex EDM (Coltene Whaledent, Cuyahoga Falls, OH) is manufactured using the technique of electrical discharge machining (EDM). EDM is a noncontact thermal erosion process used to machine electrically conductive materials using controlled electrical discharges (Jameson, 2001). The electrical sparks cause a local melting and partially evaporation of small portions of material that are removed from this local area living a typical crater-like surface finish (Theisen, 2004). After cutting cleaning is accomplished through ultrasonics in an acid bath. The instrument is heat treated at temperature ranging between 300-600 ˚C for 10 min to 5 hours before or after the cleaning process (Pernot, 2015). EDM files have Af temperatures over 52 °C (Iacono, 2016). Additionally, the EDM process produces a non-directional surface finish, thereby avoiding inducement of early material failure that results from conventional grinding techniques (Carlos, 2004). XP endo shaper (FKG Dentaire, La-Chaux-de-Fonds, Switzerland) is introduced in 2016 with 0.30 diameter and 0.1 taper that could expend to 0.4 taper (XP endo Shaper, 2016). This file has a retracted form to rectilinear geometry when it is in a martensitic phase (rest position or static) and a structured form when in the austenite phase (working position or dynamic state). The transition from the martensite phase to the austenite phase occurs naturally in the body temperature between 32 ° C and 37 ° C with Af temperature around 35° C. In dynamic state, the instrument has a twisted shape, with several twists twisted along its length (Rouiller, 2014). K3 XF (SybronEndo, Orange, CA, USA) is a grinded file, submitted to heat treatment in order to enhance flexibility and resistance to cyclic fatigue due to the proprietary R-phase technology (Gambarini, 2011 and Ha, 2013). K3XF instruments have an Af temperature below 37˚C. Therefore, it has an austenite structure at body temperature and would exhibit a super elastic property during clinical application. The heat treatment processing used for K3XF, may modify the transformation temperature by releasing crystal lattice defects and diminishing internal strain energy. On the other hand, 2 overlapping endothermic peaks were observed on the heating plot indicating that reverse transformation of the alloy passes through the intermediated R-phase, which reflected the complex phase transformation behavior tracking back to the manufacturing process (Shen, 2013).

Effect of heat treatment on cyclic fatigue

Cyclic fatigue resistance of rotary instruments is determined by cross-sectional design, surface condition (roughness and residual stress), and thermomechanical process applied during manufacturing (Park, 2010 and Dalibor vojtech, 2010). The correlation between martensitic transformation temperatures and mechanical properties has been extensively discussed. Dalibor (Dalibor Vojtech, 2010), found that all annealing temperatures result in TS reduction (stress at the onset of B2→ B19′ transformation) except for a very short annealing at 410°C. The rate of TS reduction increases with increasing temperature. This was also explained by the precipitation of Ni-rich Ti3Ni4 particles from the NiTi matrix which leads to a nickel depletion in the matrix, resulting in an increase of transformation temperatures and in a proportional decrease of the transformation stress, according to the Clausius-Clapeyron relationship (Filip, 2001). Miyai (Miyai, 2006), Stated that lower Ms impedes the phase transformation and more stress is required to induce martensitic transformation. When the working environment is below the AF temperature, the Nitinol microstructurecomprises martensite which exhibits higher flexibility and lower stiffness than Austenite. The lower stiffness of martensitic instruments can be attributed to the lower Young's modulus of martensite (about 30-40 GPa) whereas austenite is about (80-90 GPa) at ambient temperature (Johnson, 2014). Instruments with the martensitic microstructure have shown 23% reduction in bending torque compared to SE NiTi. The more martensitic the NiTi alloy is the more flexible and fatigue resistant an instrument becomes (Santoro, 2001). This could be explained by the fact that the martensitic NiTi wire allows a greater amount of deformation at a similar torque than austenitic NiTi alloy (Park, 2010 and Johnson, 2014). The second generation has enhanced resistance to cyclic fatigue. Studies have been performed using either static (Testarelli, 2009; Gambarini, 2008; de Vasconcelos, 2016) or dynamic (Haïkel, 1999; Kramkowski, 1999; Johnson, 2008 and Gao, 2010).Testing protocols conducted at room or body temperature. The instruments made from M-Wire with a ProFile design exhibited superior resistance to cyclic fatigue than SE wire instruments of the same size (Johnson, 2008). GTX manufactured with M-Wire are more resistant to flexural fatigue than are similar GT instruments made with conventional NiTi wire, mainly because of the thermomechanical treatment (da Cunha Peixoto, 2010).

ProFile Vortex had a significantly higher cyclic fatigue life (150% better) compared with SE-wire (Kramkowski, 2009). TF showed increased cyclic fatigue resistance compared to K3 (Kim, 2010). TYP CM, file was shown to be 300% more resistant to fatigue failure than TYP instrument (Shen, 2011). It is consistent with previous studies that found that instruments from CM wires were significantly more resistant to fatigue failure than those of SE-wires (Shen, 2012; Plotino, 2014).

The Third generation of Niti has shown an increase of cyclic fatigue resistance compared to first and second:

Gambarini *et al.* (2011), Ha *et al* (2013), found that K4 prototype or K3XF instruments showed an increase in the mean number of cycles to failure when compared with K3 instruments.PTG files were significantly more flexible and resistant to fatigue than PU files (Hieawy, 2015), and demonstrated the highest performance in terms of cyclic fatigue resistance when compared to PTN and PU (Uygun, 2016). Vortex Blue was ranked first in both fatigue life and flexibility followed by M-Wire, SE wire, and stainless steel files (Gao, 2012). Hyflex EDM showed a higher cyclic fatigue resistance than Reciproc and Wave One (Pedullà, 2016). De Vasconcelos *et al.* (2016), found that the more martensitic NiTi alloy is the more flexible and the more fatigue resistant an instrument becomes likely because of thermal treatment in the creation of the alloy that produces a better arrangement of the crystal structure and changes in the relative percentage of phases present in the alloy. These results corroborates findings of Santoro *et al* (2001), Shen *et al* (Shen, 2013), Grande *et al* (Haïkel, 1999). Other possible reasons for fatigue resistance is that the martensitic phase transformation has damping characteristics, because of the energy absorption characteristics of its twinned phase structure, which render crack propagation more difficult because of the larger number of interfaces present (Thompson, 2000). A complex array of secondary cracks is formed because of these interfaces, dissipating the energy required for crack propagation (Pereira, 2013).It might be also that Fatigue-crack growth resistance of the Martensite is superior to Austenite (McKelvey, 2001). Moreover, other yet unknown factors may play a role in reducing fatigue resistance. (de Vasconcelos, 2016). Therefore, it is now admitted that heat treatment has a positive impact on enhancing fatigue resistance.

Effect of heat treatment on torsional resistance properties

Torsional resistance is mainly affected by several factors including metal mass, cross-sectional design, alloy properties and the presence of defects associated with the manufacturing process (Berutti, 2003). Reports have been conflicting regarding the influence of heat treatment on torsional fatigue.

In the first study on this subject, Johnson *et al* showed that the torque at fracture of ProFile 25/.04 made from M-Wire was similar to that of existing ProFile files made from SE wire (Johnson, 2008). Kramkowski and Bahcall (Kramkowski, 2009), also found that there was no statistical difference when comparing the torque (g/cm) required to induce a torsional failure of ProFile GT and ProFile GTX files of identical file sizes. However, the angle of deflection of ProFile GT was significantly greater before separation than that of ProFile GTX for all file sizes tested except for 20/.04. The torque and angle of rotation are reported to give valuable information about the torsional fracture of an instrument when a bound file tip is rotated. It was found that as tip size and taper of instruments increased the torque at fracture also increased.

Gao *et al.* (Gao, 2010), studied the torsional properties of SE wire files, M-Wire Files, and Vortex Blue files. Results showed that M-Wire held a slight edge over Vortex Blue NiTi in torsional strength. SE wire and Vortex Blue displayed the same average peak torque. Vortex Blue showed the greatest distortion angle at break, whereas SE wire and M-Wire showed no significant differences in the degree of rotation (Seto, 1999). M-wire technology has been shown to increase the resistance to flexural fatigue but lower the torsional strength with an elongated fracture angle (da Cunha Peixoto, 2010). The torsional resistance of GTX was lower than that of similar GT instruments (da Cunha Peixoto, 2010). Kwak *et al* (Kwak, 2016), evaluated the effect of heat treatment on the mechanical properties of the glide path preparation instruments. The torsional strengths and toughness of the files were reduced by heat treatment. However, the screw-in test revealed that heat-treated files had a significantly lower screw-in effect(Kwak, 2016). Rotary files tend to thread and screw into root canals, which subjects them to high levels of stress as they bind and lock in the canal (Ha, 2015). Yum *et al* (Yum, 2011), and park *et al* (Park, 2010), compared torsional strength, distortion angle, and toughness of various nickel-titanium (NiTi) rotary files to study their mechanical behavior under torsional stress. The TF presented the lowest torsional resistance in these studies. One possible explanation is that an instrument made from the R-phase alloy would be more flexible, allowing a greater amount of deformation at a similar torque than austenitic NiTi.

Hyflex instruments made of CM wire showed similar torsional resistance compared to SE NiTi (Peters, 2012). Its maximum torsional strength was lower than Reciproc and WO, whereas no significant difference was found comparing these reciprocating instruments to each other's. However, HEDM showed significantly higher angular rotation to fracture (and therefore significantly higher time before torsional fracture) (Pedullà, 2016). The results showed that M-wire instruments, such as Reciproc and WO, generally possess greater torque resistance but smaller angles of rotation before fracture than CM-wire files such as HEDM. Posts machining thermal-treated K3XF instruments maintain the same torsional properties as conventional K3 instruments. (Kramkowski, 2009). Interestingly, in the SEM analysis of the fracture surface (Ha, 2013), K3XF showed numerous micropores with various diameters on the side aspect of the file flute. These pores seem to limit crack propagation, probably acting as a stopper and/or distributor. On the side surface of K3XF near the torsional fracture area, the micropores were compressed and deformed elliptically. With this hypothetical mechanism, the stiffness of the instrument seems to be decreased, and its flexibility is increased. Shen *et al* explored the impact of preloading of torsional angular deformation on the cyclic fatigue resistance as well as the impact of recycled fatigue on the torsional resistance of conventional superelastic K3 and heat-treated K3XF instruments (Shen, 2015). Only 75% fatigue preloading reduced the torsional strength and distortion angle of K3 instruments. Cyclic fatigue had no effect on the torsional fracture resistance of K3XF files. Therefore, it is expected that thermomechanically treated NiTi instruments maintain the same torsional properties as conventional NiTi instruments.

Conclusion

The changes in the mechanical behavior of NiTi instruments due to thermal processing is well documented. Heat treated Niti files would essentially be in the martensite /R phase state in working environment and /or body temperature. This enhancement offered promising results, concerning the cyclic resistance, yet the impact on torsional failure have not been conclusive. Furthermore, it opened a totally new era in file designing and concepts capitalizing on stress induced martensite behavior and leading to new file systems. The thermomechanical treatment seems to be a predictable method of gaining considerable benefits by enhancing the efficiency, safety and performance of endodontic files.

REFERENCES

Alapati SB, Brantley WA, Iijima M, Schricker SR, Nusstein JM, Li U-M, *et al.* Micro-XRD and temperature-modulated DSC investigation of nickel-titanium rotary endodontic instruments. Dent Mater Off Publ Acad Dent Mater. 2009 Oct;25(10):1221–9.

Alapati, S.B., Brantley, W.A., Iijima, M., Clark, W.A.T., Kovarik, L., Buie C, *et al.* Metallurgical characterization of a new nickel-titanium wire for rotary endodontic instruments. *J Endod.* 2009 Nov;35(11):1589–93.

Ammon D, Shotton V, Gao Y, Maxwell R. Endodontic instruments and methods of manufacturing thereof. US Patent Application 8916009 B2, 2014.

Berendt C. Method of preparing Nitinol for use in manufacturing instruments with improved fatigue resistance. US Patent Application 20070072147, 2007.

Berutti E, Chiandussi G, Gaviglio I, Ibba A. Comparative analysis of torsional and bending stresses in two mathematical models of nickel-titanium rotary instruments: ProTaper versus ProFile. J Endod. 2003 Jan;29(1):15–9.

Brantley W, B. S. Heat Treatment of Dental Alloys: A Review. In: Pardhi Y, editor. Metallurgy - Advances in Materials and Processes [Internet]. InTech; 2012 [cited 2016 Sep 7].

Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH. Differential scanning calorimetric studies of nickel titanium rotary endodontic instruments. J Endod. 2002 Aug;28(8): 567–72.

Brantley WA, Svec TA, Iijima M, Powers JM, Grentzer TH. Differential scanning calorimetric studies of nickel-titanium rotary endodontic instruments after simulated clinical use. J Endod. 2002 Nov;28(11):774–8.

Carlos A. Aloise, Gary T. Garman. Method of manufacturing an endodontic instrument. US Patent Application 6783438 B2, 2004.

da Cunha Peixoto IF, Pereira ESJ, da Silva JG, Viana ACD, Buono VTL, Bahia MG de A. Flexural fatigue and torsional resistance of ProFile GT and ProFile GT series X instruments. J Endod. 2010 Apr;36(4):741–4.

Dalibor vojtech. Influence of heat treatment of shape memory NiTi alloy on its mechanical properties. Roznov Pod Radhostem Czech Repub EU Met. 2010 5;(18).

de Vasconcelos RA, Murphy S, Carvalho CAT, Govindjee RG, Govindjee S, Peters OA. Evidence for Reduced Fatigue Resistance of Contemporary Rotary Instruments Exposed to Body Temperature. J Endod. 2016 Mar 15;

Filip P, Mazanec K. On precipitation kinetics in TiNi shape memory alloys. Scr Mater. 2001 Sep;45(6):701–7.

Gambarini G, Grande NM, Plotino G, Somma F, Garala M, De Luca M, *et al.* Fatigue resistance of engine-driven rotary nickel-titanium instruments produced by new manufacturing methods. J Endod. 2008 Aug;34(8):1003–5.

Gambarini G, Plotino G, Grande NM, Al-Sudani D, De Luca M, Testarelli L. Mechanical properties of nickel-titanium rotary instruments produced with a new manufacturing technique. Int Endod J. 2011 Apr;44(4):337–41.

Gao Y, Gutmann JL, Wilkinson K, Maxwell R, Ammon D. Evaluation of the impact of raw materials on the fatigue and mechanical properties of ProFile Vortex rotary instruments. J Endod. 2012 Mar;38(3):398–401.

Gao Y, Maxwell R. Endodontic rotary instruments made of shape memory alloys in their martensitic state and manufacturing methods. US Patent Application 201102 71529 A1, 2011.

Gao Y, Shotton V, Wilkinson K, Phillips G, Johnson WB. Effects of raw material and rotational speed on the cyclic fatigue of ProFile Vortex rotary instruments. J Endod. 2010 Jul;36(7):1205–9.

Grande NM, Plotino G, Pecci R, Bedini R, Malagnino VA, Somma F. Cyclic fatigue resistance and three-dimensional analysis of instruments from two nickel-titanium rotary systems. Int Endod J. 2006 Oct;39(10):755–63.

Gutmann JL, Gao Y. Alteration in the inherent metallic and surface properties of nickel-titanium root canal instruments to enhance performance, durability and safety: a focused review. Int Endod J. 2012 Feb;45(2):113–28.

Ha JH, Cheung GSP, Versluis A, Lee CJ, Kwak SW, Kim HC. “Screw-in” tendency of rotary nickel-titanium files due to design geometry. Int Endod J. 2015 Jul;48(7):666–72.

Ha J-H, Kim SK, Cohenca N, Kim H-C. Effect of R-phase heat treatment on torsional resistance and cyclic fatigue fracture. J Endod. 2013 Mar;39(3):389–93.

Haïkel Y, Serfaty R, Bateman G, Senger B, Allemann C. Dynamic and cyclic fatigue of engine-driven rotary nickel-titanium endodontic instruments. J Endod. 1999 Jun;25(6):434–40.

Hieawy A, Haapasalo M, Zhou H, Wang Z, Shen Y. Phase Transformation Behavior and Resistance to Bending and Cyclic Fatigue of ProTaper Gold and ProTaper Universal Instruments. J Endod. 2015 Jul 1;41(7):1134–8.

Hou X, Yahata Y, Hayashi Y, Ebihara A, Hanawa T, Suda H. Phase transformation behaviour and bending property of twisted nickel-titanium endodontic instruments. Int Endod J. 2011 Mar;44(3):253–8.

Iacono F, Pirani C, Generali L, Bolelli G, Sassatelli P, Lusvarghi L, *et al.* Structural analysis of HyFlex EDM instruments. Int Endod J. 2016 Feb 11;

Jameson, E.C. Description and Development of EDM, Electrical Society of Manufacturing Engineers, Dearbern, Michigan, pp16, 2001.

Johnson E, Lloyd A, Kuttler S, Namerow K. Comparison between a novel nickel-titanium alloy and 508 nitinol on the cyclic fatigue life of ProFile 25/.04 rotary instruments. J Endod. 2008 Nov;34(11):1406–9.

Johnson WB. Fatigue-Resistant Nitinol Instrument. US Patent Application 20140242543 A1, 2014.

Johnson WB. Multi-Planar Pre-Curved Rotary Endodontic File. US Patent Application 20130189644 A1, 2013.

Jordan L, Sultan A, Vermaut P. Microstructural and mechanical characterizations of new Ni-Ti endodontic instruments. Schryvers N, Van Humbeeck J, editors. MATEC Web Conf. 2015;33:03005.

Kell T, Azarpazhooh A, Peters OA, El-Mowafy O, Tompson B, Basrani B. Torsional profiles of new and used 20/.06 GT series X and GT rotary endodontic instruments. J Endod. 2009 Sep;35(9):1278–81.

Kim H-C, Yum J, Hur B, Cheung GS-P. Cyclic fatigue and fracture characteristics of ground and twisted nickel-titanium rotary files. J Endod. 2010 Jan;36(1):147–52.

Kramkowski TR, Bahcall J. An in vitro comparison of torsional stress and cyclic fatigue resistance of ProFile GT and ProFile GT Series X rotary nickel-titanium files. J Endod. 2009 Mar;35(3):404–7.

Kwak SW, Ha J-H, Lee C-J, El Abed R, Abu-Tahun IH, Kim H-C. Effects of Pitch Length and Heat Treatment on the Mechanical Properties of the Glide Path Preparation Instruments. J Endod. 2016 Mar 10;

Liu J. Characterization of New Rotary Endodontic Instruments Fabricated from Special Thermomechanically Processed NiTi Wire [Internet]. The Ohio State University; 2009 [cited 2016 May 10]. Available from: https://etd.ohiolink.edu/pg_10?0::NO:10:P10_ACCESSION_NUM:osu1244643081

McKelvey AL, Ritchie RO. Fatigue-crack growth behavior in the superelastic and shape-memory alloy nitinol. Metall Mater Trans A. 2001 Mar; 32(13):731–43.

Miyai K, Ebihara A, Hayashi Y, Doi H, Suda H, Yoneyama T. Influence of phase transformation on the torsional and bending properties of nickel-titanium rotary endodontic instruments. Int Endod J. 2006 Feb;39(2):119–26.

Otsuka K, Ren X. Physical metallurgy of Ti–Ni-based shape memory alloys. Prog Mater Sci. 2005 Jul;50(5):511–678.

Park S-Y, Cheung GSP, Yum J, Hur B, Park J-K, Kim H-C. Dynamic torsional resistance of nickel-titanium rotary instruments. J Endod. 2010 Jul;36(7):1200–4.

Pedullà E, Savio FL, Boninelli S, Plotino G, Grande NM, Rosa GL, *et al.* Torsional and Cyclic Fatigue Resistance of a New Nickel-Titanium Instrument Manufactured by Electrical Discharge Machining. J Endod. 2016 Jan 1;42(1):156–9.

Pereira ESJ, Gomes RO, Leroy AMF, Singh R, Peters OA, Bahia MGA, *et al.* Mechanical behavior of M-Wire and conventional NiTi wire used to manufacture rotary endodontic instruments. Dent Mater Off Publ Acad Dent Mater. 2013 Dec;29(12):e318–24.

Pereira ESJ, Peixoto IFC, Viana ACD, Oliveira II, Gonzalez BM, Buono VTL, *et al.* Physical and mechanical properties of a thermomechanically treated NiTi wire used in the manufacture of rotary endodontic instruments. Int Endod J. 2012 May 1;45(5):469–74.

Pernot J, Rolland X, Euvrard H. Endodontic instrument with rough surfaces, and method for producing such an instrument. Patent WO2015028743 A1, 2015.

Peters OA, Arias A, Paqué F. A Micro-computed Tomographic Assessment of Root Canal Preparation with a Novel Instrument, TRUShape, in Mesial Roots of Mandibular Molars. J Endod. 2015 Sep;41(9):1545–50.

Peters OA, Gluskin AK, Weiss RA, Han JT. An in vitro assessment of the physical properties of novel Hyflex nickel-titanium rotary instruments. Int Endod J. 2012 Nov;45(11):1027–34.

Plotino G, Testarelli L, Al-Sudani D, Pongione G, Grande NM, Gambarini G. Fatigue resistance of rotary instruments manufactured using different nickel-titanium alloys: a comparative study. Odontol Soc Nippon Dent Univ. 2014 Jan;102(1):31–5.

Rouiller J-C. Endodontic instrument for drilling the root canals of a tooth. US Patent Application 20140004479 A1, 2014.

Santoro M, Nicolay OF, Cangialosi TJ. Pseudoelasticity and thermoelasticity of nickel-titanium alloys: A clinically oriented review. Part I: Temperature transitional ranges. Am J Orthod Dentofacial Orthop. 2001 Jun 1;119(6):587–93.

Santos L de A, Bahia MG de A, de Las Casas EB, Buono VTL. Comparison of the mechanical behavior between controlled memory and superelastic nickel-titanium files via finite element analysis. J Endod. 2013 Nov;39(11):1444–7.

Seto BG, Nicholls JI, Harrington GW. Torsional properties of twisted and machined endodontic files. J Endod. 1990 Aug;16(8):355–60.

Shen Y, Coil JM, Zhou H, Zheng Y, Haapasalo M. HyFlex nickel-titanium rotary instruments after clinical use: metallurgical properties. Int Endod J. 2013 Aug;46(8):720–9.

Shen Y, Qian W, Abtin H, Gao Y, Haapasalo M. Effect of environment on fatigue failure of controlled memory wire nickel-titanium rotary instruments. J Endod. 2012 Mar;38(3):376–80.

Shen Y, Qian W, Abtin H, Gao Y, Haapasalo M. Fatigue testing of controlled memory wire nickel-titanium rotary instruments. J Endod. 2011 Jul;37(7):997–1001.

Shen Y, Riyahi AM, Campbell L, Zhou H, Du T, Wang Z, *et al.* Effect of a combination of torsional and cyclic fatigue preloading on the fracture behavior of K3 and K3XF instruments. J Endod. 2015 Apr;41(4):526–30.

Shen Y, Zhou H, Coil JM, Aljazaeri B, Buttar R, Wang Z, *et al.* ProFile Vortex and Vortex Blue Nickel-Titanium Rotary Instruments after Clinical Use. J Endod. 2015 Jun;41(6):937–42.

Shen Y, Zhou H, Wang Z, Campbell L, Zheng Y, Haapasalo M. Phase Transformation Behavior and Mechanical Properties of Thermomechanically Treated K3XF Nickel-Titanium Instruments. J Endod. 2013 Jul 1;39(7):919–23.

Shen Y, Zhou H, Zheng Y, Campbell L, Peng B, Haapasalo M. Metallurgical Characterization of Controlled Memory Wire Nickel-Titanium Rotary Instruments. J Endod. 2011 Nov 1;37(11):1566–71.

Shen Y, Zhou H, Zheng Y, Peng B, Haapasalo M. Current challenges and concepts of the thermomechanical treatment of nickel-titanium instruments. J Endod. 2013 Feb;39(2):163–72.

Testarelli L, Grande N., Plotino G, Lendini M, Pongione G, Paolis GD, *et al.* Cyclic Fatigue of Different Nickel-Titanium Rotary Instruments: A Comparative Study. Open Dent J. 2009 Apr 16;3:55–8.

Testarelli L, Plotino G, Al-Sudani D, Vincenzi V, Giansiracusa A, Grande NM, *et al.* Bending properties of a new nickel-titanium alloy with a lower percent by weight of nickel. J Endod. 2011 Sep;37(9):1293–5.

Theisen W, Schuermann A. Electro discharge machining of nickel–titanium shape memory alloys. Mater Sci Eng A. 2004 Jul;378(1-2):200–4.

Thompson SA. An overview of nickel-titanium alloys used in dentistry. Int Endod J. 2000 Jul;33(4):297–310.

Uygun AD, Kol E, Topcu MKC, Seckin F, Ersoy I, Tanriver M. Variations in cyclic fatigue resistance among ProTaper Gold, ProTaper Next and ProTaper Universal instruments at different levels. Int Endod J. 2016 May 1;49(5):494–9.

Walia HM, Brantley WA, Gerstein H. An initial investigation of the bending and torsional properties of Nitinol root canal files. J Endod. 1988 Jul;14(7):346–51.

Wu SK, Lin HC, Chou TS. A study of electrical resistivity, internal friction and shear modulus on an aged Ti49Ni51 alloy. Acta Metall Mater. 1990;38:95–102.

XP endo Shaper | FKG Dentaire, 2016 [Internet]. [cited 2016 Oct 3]. Available from: http://www.fkg.ch/products/endodontics/root-canal-preparation-and-retreatment/xp-endo-shaper

Xu X, Eng M, Zheng Y, Eng D. Comparative study of torsional and bending properties for six models of nickel-titanium root canal instruments with different cross-sections. J Endod. 2006 Apr;32(4):372–5.

Yum J, Cheung GS-P, Park J-K, Hur B, Kim H-C. Torsional strength and toughness of nickel-titanium rotary files. J Endod. 2011 Mar;37(3):382–6.

Zhou H-M, Shen Y, Zheng W, Li L, Zheng Y, Haapasalo M. Mechanical properties of controlled memory and superelastic nickel-titanium wires used in the manufacture of rotary endodontic instruments. J Endod. 2012 Nov;38(11):1535–40.

****