Alteration in the inherent metallic and surface properties of nickel–titanium root canal instruments to enhance performance, durability and safety: a focused review

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Abstract


The expanded use of nickel–titanium (NiTi) rotary instruments in root canal procedures has led to the development of a wide variety of shapes, designs and applications. Root canal anatomy has not changed, however, and the same challenges exist in both initial treatment and the revision of unacceptable treatment. These challenges include application with high levels of achievement and low to no levels of adverse effects, such as instrument fracture, root canal wall ledging, dentine wall perforation and so forth. To that end, many manufacturers have been seeking ways to alter the presently available and wide range of root canal instrument designs, with a focus on altering the surface of the alloy or altering the alloy microstructure with post-machining or post-twisting heat treatment. This focused review will address the impact that these modifications have had on instrument flexibility, resistance to cyclic fatigue and cutting efficiency.

Keywords: cyclic fatigue, heat treatment, NiTi, root canal instruments, surface modification instruments.

Introduction

Manufacturers of nickel–titanium (NiTi) root canal instruments have been seeking ways to alter the presently available and wide range of instrument designs, with a focus on altering the surface of the alloy or altering the alloy microstructure with post-machining or post-twisting heat treatment. This focused review will address the impact that these modifications have had on instrument flexibility, resistance to cyclic fatigue and cutting efficiency for the clinician.

Literature search strategy

The prime, refereed journals of endodontics, dental materials and materials science (Journal of Endodontics, International Endodontic Journal, Oral Surgery Oral Medicine Oral Pathology Oral Radiology and Endodontology, Dental Materials, Dental Materials Journal and Journal of Materials Science: Materials in Medicine) were hand-reviewed for the years 2005 to 2011 (64 articles were identified), and within these published articles, the appropriately identified MeSH terms were chosen using PubMed and consolidated for this review (Table 1). These terms were then used in an Ovid search, which resulted in an additional, but limited identification of articles. All articles identified had their references screened for appropriateness and duplications, with further publications being identified through hand-reviewing relative to the purpose of this
Table 1 MeSH terms chosen for refined literature search

| Dental alloys/chemistry and analysis |
| Dental polishing                      |
| Dental stress analysis                |
| Root canal preparation/instrumentation |
| Nickel and nickel analysis            |
| Titanium/analysis and chemistry       |
| Surface properties                    |
| Mechanical stress                     |
| Materials testing                     |
| Spectrum analysis                     |
| Electrochemical techniques            |
| Torsion/mechanical and rotational     |
| Heat treatment                        |
| Cold treatment                        |

review, i.e. modifications of the contemporary endodontic NiTi rotary instrument, in various ways, whether in its inherent structure or surface modifications, to enhance its resistance to cyclic fatigue, or increase or alter its flexibility and cutting efficiency.

Perspectives on the uniqueness of the nickel–titanium alloys

Shape-memory alloys (SMA) have a unique capability to recover their original shape after undergoing large deformation through heating (known as shape-memory effect, SME). The concept of SME was first described by Ölander (1932) during his evaluation of a cadmium–gold alloy. NiTi, known as Nitinol, was discovered by chance by Buchler & Wang (1963) whilst searching for nonmagnetic, salt-resistant, waterproof alloys for navel use. Besides SMEs, NiTi exhibits superelastic (SE) behaviours that allow them to return to their original shape upon unloading following substantial deformation. Superalasticity occurs in association with reversible phase transformation between austenite and martensite, so the transformation temperatures and behaviour of NiTi have a critical influence on the mechanical properties, which can be readily altered by small changes in composition, impurities and heat treatments (Yoneyama & Kobayashi 2009). This distinct property of NiTi alloys has created a revolution in the manufacture of endodontic, intracanal instruments, which has improved the speed and efficiency of intracanal procedures.

Whilst there are many alloys that have SE properties (Civjan et al. 1975), NiTi has a high level of biocompatibility along with excellent corrosion resistance (Darabara et al. 2004) under certain conditions or with some endodontic instruments, but not with others (Stokes et al. 1999, de Castro Martins et al. 2006, Ormiga Galvão Barbosa et al. 2007, Topuz et al. 2008). For example, during extended periods of time in sodium hypochlorite (NaOCl) solutions, corrosion may be enhanced (O’Hoy et al. 2003) or minimized depending on the pH of the environment (Nóvoa et al. 2007). In solutions of fluoride or acidic solutions, corrosion resistance has been identified with NiTi wires (Benyahia et al. 2009, Lee et al. 2010).

Known as nitinol in the United States, this alloy was manufactured in Shanghai, China, in 1979 as nitalloy (56 wt. % of nickel and 44 wt. % of titanium) (Yang et al. 1982). In some NiTi alloys, a small percentage (<2 wt. %) of nickel can be substituted by cobalt (Thompson 2000).

Clinicians and biomaterial scientists have been investigating the application of NiTi in medicine for a number of years (Baumgart et al. 1980). Schettler (1979) described its use in a new form of fixation for alveolar fractures, whilst many other investigators addressed its use in orthopaedic applications (Dai 1983, Yang et al. 1987). Hsieh & Yu (1982) implanted nitalloy wires and cubes in the periosteum, muscles and subcutaneous tissues of dogs and found no adverse, macroscopic tissue reactions. Microscopically, there was mild inflammation with some evidence of hyaline degeneration. Dotter et al. (1983) and Cragg et al. (1983) investigated opening blockages in blood vessels using nitinol stents that have since revolutionized the treatment for vascular disease.

The application of NiTi in endodontics was reported first by Walia et al. (1988) who used nitinol orthodontic wire (Andreasen & Morrow 1978) to fabricate intracanal files, size No. 15. These were shown to have 2–3 times the elastic flexibility in bending and torsion, as well as superior resistance to torsional fracturing when compared to similar stainless steel instruments. As early as 1992, NiTi root canal instruments were introduced into a university teaching programme (Serene et al. 1995). Clinically, the use of the NiTi intracanal instruments offered the advantages of less canal transportation during application (Bergmans et al. 2001, Gergi et al. 2010) whilst reducing treatment time.

Properties of nitinol in clinical applications

Nitinol belongs to a category of alloys referred to as ‘shape-memory alloys’ that exhibit a wide range of qualities making them useful for root canal procedures. With the advent of engine-driven, rotary NiTi
instruments, minimal forces are required to shape the root canal whilst minimizing or eliminating canal transportation. Additionally, these instruments create a more centred canal preparation, although deviations may be seen with some instruments (Bergmans et al. 2001, Schäfer & Vlassis 2004, Gergi et al. 2010). Furthermore, the degree of canal deviation or lack of centring ability of the instrument, if it should occur and result in canal deviation, is limited and relatively minor (Poulsen et al. 1995, Knowles et al. 1996, Gluskin et al. 2001, Versümer et al. 2002, Schäfer & Vlassis 2004, Taşdemir et al. 2005, Schäfer et al. 2003, Schäfer et al. 2006); however, with some instrument types and their application in canals that exhibit sharp curvatures, canal aberrations in shaping may be more pronounced (Thompson & Dummer 1998, Bergmans et al. 2001, Hülsmann et al. 2003). On the negative side, NiTi root canal instruments do not permit anticurvature filing and cannot be curved or pre-bent easily prior to use. These latter properties, however, are presently being investigated by multiple dental manufacturers.

Because the SE property of NiTi instruments diminishes the connection between cross section and instrument stiffness, files with larger or tapered diameters became possible. This resulted in the manufacture of instruments with 0.02, 0.04, 0.06, 0.08, 0.10 and 0.12 tapers. Whilst the application of these instruments has enabled a greater removal of the pulp tissue and necrotic debris and enhanced the machining of dentine (Tucker et al. 1997, Young et al. 2007), their ability to achieve thorough canal cleanliness may still be questioned (Bergmans et al. 2001). Although rotary NiTi instruments have created root canal shapes that are highly conducive to contemporary irrigation protocols, particularly in the removal of debris from anatomical irregularities and cleaning in the apical third of the canal (Bergmans et al. 2001, Tan & Messer 2002), studies have still not been able to show thorough cleanliness (Peters et al. 2001, Rödig et al. 2002, Hülsmann et al. 2003, Paqué et al. 2005, Nordmeyer et al. 2011) even whilst using appropriate irrigation protocols (Arruda et al. 2009).

Along with increased tapers, NiTi instrument have undergone a revolution regarding different designs for the cutting blades (sharp angles versus radial lands), variations in helical angles, numbers of flutes, cross-sectional configurations and tip designs. This has resulted in variations in instrument rake angle, degree of twisting or cutting, groove spacing and the availability of cutting, partial cutting and noncutting tips. The sum total of these variations seems to be focused on an instrument that is highly efficient in its cutting ability whilst exhibiting resistance to fracture during its application in even the most difficult anatomical confines. Even with these innovations, however, instrument fracture is a main concern amongst clinicians and can occur without visible signs of previous permanent deformation, even when used within the elastic limit of the instrument (Parashos & Messer 2004, 2006a).

Two additional factors may impact on cutting efficiency, those being the rake angle and the depth of the groove or flute at the base of the cutting edge (Bergmans et al. 2001, Peters 2004, Hülsmann et al. 2005, Metzger et al. 2011). Most rotary NiTi instruments have either neutral cutting angles or active cutting blades with positive cutting angles (Schäfer & Oitzinger 2008). Instruments with actively cutting blades have been shown to result in better canal cleanliness (Hülsmann et al. 2005). With regard to the instrument’s groove, a deeper groove promotes more dentine chip removal capability (Schäfer 1999) and is related to enhanced instrument cutting efficiency (Bergmans et al. 2001).

Despite the increase in instrument flexibility and cutting efficiency, possibly the biggest deterrent to the clinical adoption of NiTi instruments is the fear of instrument fracture (Kuhn et al. 2001, Bergmans et al. 2001, Parashos & Messer 2006a,b, Larsen et al. 2009). The most important factor that leads to separation is metal fatigue. This is especially predominant when working in curved root canals, where half of the instrument is in tension (outside of the curve) and the other half in compression (inside of the curve) with stresses being the greatest in the curvature (Pruett et al. 1997).

The second factor that may impact on instrument separation is torsional loading (referred also as torsional fracture or resistance). This parameter of challenge to NiTi instruments is usually evaluated with a torsionmeter, where the fracture of the instrument as a result of a torsional overload is expressed as the maximum torque and the angular distortion at separation (Yum et al. 2011). This outcome is usually dependent on instrument design and manipulation in the canal, the latter of which reflects the fundamental mechanical behaviour during loading and is usually operator-dependent. However, even if instrument fracture does not occur because of torsional overloading, the instrument will have a mean number of cycles to failure that is determined by specific parameters of canal radius, canal angle, instrument diameter and...
other inherent factors in the metal itself (Pruett et al. 1997). Additionally, the use of higher rpm (which is standard in the present generation of NiTi instruments – 300 to 500+ rpm) will consume the useful life of the instrument (Lopes et al. 2009), again making a case for a single-use tool. The reader is referred to extensive publications by Peters (2004), Hülsmann et al. (2005), and Peters & Paqué (2010) for reviews of specific rotary NiTi root canal instruments, along with comparative reviews of their performances as discussed in the literature.

With all due respect for the positive attributes of the contemporary NiTi intracanal instrument, there is a need to develop instruments with greater flexibility, increased resistance to cyclic fatigue and great cutting efficiency. These issues focus primarily on overall instrument efficiency whilst decreasing or eliminating the concern for fracture during usage (Tripi et al. 2006). To this end, various approaches to alterations in the way the instruments are manufactured or the alteration in the physical properties of NiTi instruments have attracted the interest and focus of manufacturers and scientists alike. Likewise, the surface condition of the NiTi instrument is a critical factor that influences fatigue resistance because most fatigue failures nucleate at the surface, especially in the presence of high stress amplitude or surface defects (Bahia & Buono 2005). Shabalovskaya et al. (2008) published a critical overview on Nitinol surfaces and their modifications for medical applications. They presented a comprehensive analysis of surface topography, chemistry, corrosion behaviour, nickel release and biological responses to Nitinol surfaces modified mechanically or using such methods as etching in acids and alkaline solutions, electropolishing, heat and ion beam treatments, boiling in water and autoclaving, conventional and ion plasma implantation, laser melting and bioactive coating deposition. However, the majority of these treatments or modifications have little to do with NiTi endodontic instruments and the uniqueness of their applications. Therefore, studies that are more focused for alterations in NiTi instruments will be addressed, which include implantation and electropolishing to correct or manage surface defects, and variations in metal processing and file manufacturing, including twisting versus machining and heat treatments. Underlying many of these processes has been the alteration in the raw Nitinol wire under various strain-annealing processes resulting in a material that includes some portion of the alloy in both the martensitic and the R-phase (an intermediate structure when moving from the austenitic phase to the martensitic phase) whilst maintaining a pseudo-elastic state (Kuhn et al. 2001, Kuhn & Jordan 2002, Johnson et al. 2008).

Surface treatment – implantation

Attempts to enhance the surface of NiTi instruments and thereby to minimize or eliminate inherent defects, increase surface hardness and increase flexibility, resistance to cyclic fatigue and cutting efficiency have resulted in a variety of strategies. Multiple studies have sought to achieve these goals by implanting argon (Ar) (Lee et al. 1996), boron (B) (Wolle et al. 2009) and nitrogen (N) (Lee et al. 1996, Rapisarda et al. 2000, Tripi et al. 2002, Gavini et al. 2010), performing thermal nitridation (Rapisarda et al. 2000) and plasma immersion (Li et al. 2007, Alves-Claro et al. 2008) or deep dry cryogenic treatment (Vinothkumar et al. 2007) without affecting the SE bulk mechanical properties of the alloy. Ion implantation (Ar and B) gave promising results with selected instruments, nitrogen and titanium nitride (TiN) gave mixed results, although recent studies have been quite favourable (Gavini et al. 2010), and plasma immersion resulted in increased wear resistance (Alves-Claro et al. 2008). Whilst deep dry cryogenic treatment increased the cutting efficiency of the instrument significantly, it did not increase the wear resistance (Vinothkumar et al. 2007).

Clinical implications

From a clinical perspective, attempts to enhance cutting efficiency and surface characteristics have not taken into account fully the potential impact that clinical sterilization may have on NiTi instrument. Studies have indicated that following sterilization, there appears to be a decrease in the cutting efficiency of the instrument, an increase in the depth of surface irregularities and surface roughness, and evidence of crack initiation and propagation (Mize et al. 1998, Alexandrou et al. 2006, Valois et al. 2008). In fact, instruments that underwent the greatest number of sterilizations showed in-depth distributions of chemical composition that were different from those seen in the control group; this was identified as being the result of greater amounts of titanium oxide on the surfaces of the sterilized instruments. These same files also showed a decrease in cutting efficiency in comparison with those of the control group (Rapisarda et al. 1999). Furthermore, newer NiTi files that have been twisted instead of
machined showed some evidence of decreased resistance to cyclic fatigue with increased, cumulative autoclaving procedures (Hiller et al. 2011).

In clinical contrast, the impact of NaOCl did not show any difference in the cutting efficiency or resistance to fracture of NiTi instruments (Ormiga Galvão Barbosa et al. 2007, Cavalleri et al. 2009), although it did result in a reduced resistance to cyclic fatigue (Berutti et al. 2006, Peters et al. 2007) and the presence of corrosion (Sonntag & Peters 2007); however, this latter result has been shown to be minimized by lowering the pH of the NaOCl to 10.1, thereby allowing the system to reach the stability domain of the passivating species TiO₂ and NiO₂ (Nóvoa et al. 2007). Studies by Oh et al. (2006) addressed the need for high corrosion resistance with NiTi alloys destined for biomedical applications. To enhance the corrosion resistance, they investigated the effect of addition of silver to NiTi alloys. Arc melting, homogenization, hot rolling and solution heat treatment were performed to prepare the nickel–titanium–silver (NiTi-Ag) specimens. The properties of the ternary NiTi-Ag alloys such as phase-transformation temperature, microstructure, microhardness, corrosion resistance and cytotoxicity were investigated. The NiTi-Ag alloys showed low silver recovery rates for the cast alloy, owing to low silver recovery rates for the cast alloy, and low silver solubility in NiTi. Silver addition to NiTi increased the transition temperature range to 100 °C and stabilized the martensitic phase at room temperature, because the starting temperature of martensitic transformation was above room temperature. Martensitic and austenitic phases existed in X-ray diffraction patterns of solution-annealed NiTi-Ag alloys. Silver addition was considered to improve the corrosion resistance and form a stable passive film. Significantly, the mechanical properties of the silver-added alloys were dependent upon the amount of alloying addition. There was no toxicity in the NiTi-Ag alloys, as the response index showed none or mild levels. Further studies in which the percentage of NaOCl was low (1%) showed no major impact on NiTi instruments; however, those that studied the impact of a 5% solution noted significant changes (Busslinger et al. 1998). Likewise, a sterilization cycle along with immersion in NaOCl did not identify any substantial instrument changes (Häikel et al. 1998). However, when NiTi instruments were coated with a physical vapour deposition of TiN, the sterilization process did not impact on the cutting efficiency nor did soaking in NaOCl (Schäfer 2002). However, studies have disputed the impact on heat sterilization and the tendency of NiTi files to lose mechanical resistance and fracture sooner during use (Silvaggio & Hicks 1997). These findings tend to portray mixed outcomes when it comes to the support for a single-use instrument vis à vis instrument sterilization and contemporary use of various percentages of NaOCl; albeit, clinical data indicate that a single use is preferred to prevent instrument fracture in the root canal, especially with smaller-sized instruments (Shen et al. 2009a,b).

**Surface treatment – electropolishing**

Many studies have investigated the impact of electropolishing on the surface condition of NiTi root canal instruments and their resistance to fracture owing to cyclic fatigue and torque (Cheung et al. 2007, Bonacorso et al. 2008a,b, Barbosa et al. 2008, Boessler et al. 2009, Shen et al. 2009c, Sinan et al. 2010, Lopes et al. 2010). Often, these studies present results that cannot be extrapolated to all NiTi instruments because of variations in the instrument designs and experimental formats and procedures (Cheung et al. 2007, Herold et al. 2007, Praisarnti et al. 2010).

Electropolishing is a method commonly used for surface finishing of many metallic medical appliances (Shabalovskaya et al. 2008). This process has been used by some companies for NiTi root canal instruments. The instruments connected to an anode are immersed together with another electrode in a temperature-controlled bath of electrolytes, followed by passing direct electrical current into the solution. The metal at the anode is dissolved into the solution, whereas a reduction reaction will occur at the cathode. This process alters the surface composition and texture of the instrument and renders a more homogeneous surface oxide layer that is a protective film, with less defects and residual surface stress. In the process, the corrosion resistance of the metal is enhanced along with improved surface characteristics.

Anderson et al. (2007) investigated the effect of electropolishing on cyclic flexural fatigue and torsional strength of three different manufacturer’s rotary NiTi endodontic instruments comparing them to nonelectropolished instruments of the same type. The number of rotations to fracture and torque at fracture were determined and compared between the instruments tested. Overall, electropolished instruments performed significantly better than nonelectropolished instruments in cyclic fatigue testing and, to a lesser extent, in static torsional loading. They concluded that the
benefits of electropolishing are likely to be caused by a reduction in surface irregularities that serve as points for stress concentration and crack initiation.

Cheung et al. (2007) compared the low-cycle fatigue (LCF) behaviour of electropolished and nonelectropolished NiTi instruments of the same design in 1.2% NaOCl solution. Number of revolutions to failure, crack-initiation sites, extent of slow crack extension into the fracture cross section, and surface-strain amplitude were noted, and a linear relationship was found between LCF life and surface-strain amplitude for both groups, with no discernible difference between the two. Although surface smoothness was enhanced by electropolishing, the instrument was not protected from LCF failure.

Barbosa et al. (2008) analysed the influence of electrochemical polishing on flexural fatigue and torsional properties of one manufacturer’s NiTi endodontic rotary instrument. New files and polished files were tested for flexural fatigue and for resistance to fracture by twisting. Statistical analysis was used to compare the groups for number of cycles, angle of rotation and maximum torque necessary to fracture. No statistical difference existed between these groups. These results suggest that electrochemical polishing has no influence on resistance to fracture of the rotary instruments tested.

Bonaccorso et al. (2008a,b) chemically analysed 36 (18 experimental/18 control) rotary NiTi instruments with and without electropolishing after cleaning procedures with NaOCl. The surface of each instrument was analysed before and after cleaning in NaOCl by using energy-dispersive X-ray analysis. After immersion in NaOCl, the nonelectropolished and electropolished files showed a significant increase in iron deposits as a result of galvanic corrosion of the shaft. The nonelectropolished files showed marked presence of NaCl deposits in the machining marks and microcracks. On the surface, the electropolished files had an oxide increase compared with the low oxide concentration (mainly TiO₂) before cleaning. The nonelectropolished files already possessed higher oxide concentration (TiO₂ and NiO) before NaOCl cleaning. NaOCl treatment affected the chemical composition of the surface and, in particular for the nonelectropolished instruments, of the bulk exposed through machining marks and fabrication microcracks.

Bui et al. (2008) investigated the effect of electropolishing NiTi rotary instruments on torque resistance, fatigue resistance and cutting efficiency. Cyclic fatigue was determined by counting rotations until breakage with an applied 30°, 45° and 60° curve with a radius of 5 mm. Statistical analysis of the data indicated that electropolishing significantly reduced resistance to cyclic fatigue but did not affect torsional resistance. However, electropolishing reduced the angle of rotation at failure and amount of unwinding. Electropolishing did not significantly affect cutting efficiency.

Boessler et al. (2009) studied the impact of two surface types, machined and electropolished, of various NiTi shaping files on torque and force during simulated root canal preparation. Pilot holes of 0.5 mm diameter were drilled perpendicularly through 3-mm-thick human dentin discs and served as standardized simulated root canals (SSRCs). Maximum torque (in Newton centimetre) and maximum force values (in Newton) were measured. In three experimental groups, preparation of SSRCs was performed using electropolished versus machined instruments in the same dentin disc. For all three tested instruments, peak torque was higher for electropolished files, with similar forces exerted apically.

Sinan et al. (2010) evaluated the effect of electropolishing time on the fracture resistance of NiTi endodontic instruments in flexion over a set time period. Each instrument was rotated at 275 rpm in a steel pipe angled at 60° with a radius of 10 mm. Electropolishing from 70 till 90 s improved the surface of the instruments but did not alter the resistance to fracture during flexion.

Praisarnri et al. (2010), using instruments that were magnetoelectropolished (Rockicki & Hryniewicz 2008), subjected them to rotational bending at various degrees of curvatures whilst immersing in 1.2% NaOCl solution until broken. The fatigue life of both nonpolished and electropolished instruments generally declined with increasing surface-strain amplitude with a resultant significant difference between the test and controlled instruments. Comparing magnetoelectropolished instruments with its commercially available nonelectropolished counterpart, an improved resistance to fatigue breakage was noted as a result of electropolishing.

Most recently, Lopes et al. (2010) found that electropolished instruments (BioRace, FKG Dentaire, La Chaux-de-Fonds, Switzerland) demonstrated significant increases in cyclic fatigue resistance. Electropolished instrument exhibited fine surface cracks that assumed an irregular or zigzag path, whilst the nonpolished files had cracks running along the machining grooves. Condorelli et al. (2010) identified the same type of increased resistance to cyclic fatigue
with the electropolished instruments (RaCe, FKG Dentaire) after performing thermal treatments. Before thermal applications, differences in the fatigue resistance were noted between the electropolished and nonelectropolished instruments that were attributed to differences in surface morphology. The thermal applications did not alter instrument surface morphology but resulted in significant changes in the instrument bulk with the appearance of an R-phase and improved fatigue resistance.

In reviewing the studies cited in this section, electropolishing appears to have a beneficial impact in that it enhances cyclic fatigue and peak torque values for NiTi instruments, the results of which may vary when the instruments are placed under significant flexion. File surfaces were smooth and improved with electropolishing, with minimal crack formation visible, thereby evidencing minimal deterioration of the instrument in the presence of NaOCl. The apparent positive effects of electropolishing, however, may vary depending on instrument type, design and, in particular, cross-sectional area (Sattapan et al. 2000, Yao et al. 2006, Oh et al. 2010).

**Twisting versus machining/grinding of the NiTi instrument**

**Twisted files**

Historically, both carbon steel and stainless steel K-files were produced by grinding graduated sizes of round wire into various shapes. This was followed by a second grinding that tapered the pieces of wire that were ultimately twisted to form a standardized 0.02-tapered instrument. However, in the 1980s, dental manufacturers began to experiment with machining stainless steel K-files, whereby the flutes were cut into a tapered, round stainless steel blank to create a similar triangular cross-sectional file instead of twisting the blank (Seto et al. 1990). Research comparing the torsional properties of the machined file with the twisted file (TF) showed that machined files exhibited less ductility than TFs prior to fracture and therefore were more susceptible to torsional failure (Seto et al. 1990).

With the advent of NiTi rotary instruments, metallic properties dictated that the metal be ground or machined to meet the desired specifications of taper, flute design, cutting edge, helical angle and so forth. If not ground, the SE NiTi blanks would return to their original shape after the release of applied forces in the twisting process; therefore, grinding was used initially with SE NiTi instruments.

In 2008, the technology for twisting NiTi was introduced (SybronEndo, Orange, CA, USA). Claims were made that a TF had unsurpassed strength, flexibility and resistance to fatigue (Gambarini et al. 2008a,b,c, 2009, Testarelli et al. 2009a,b), especially when manufactured using a treatment technology identified as R-Phase heat treatment. More specifically, TF instruments were created by taking raw NiTi wire in the austenite crystalline structure and transforming it into a different phase of crystalline structure (R-phase) by a process of heating and cooling (Gambarini et al. 2008a). As mentioned in the section ‘Properties of Nitinol in Clinical Applications’, R-phase is an intermediate structure when moving from the austenitic phase to the martensitic phase. Existence of R-phase is often identified in a differential scanning calorimetry (DSC) curve with two endothermic peaks occurred during heating cycle, corresponding to transition from martensite to R-phase (M → R) followed by transition from R-phase to austenite (R → A). However, the creation of R-phase is dependent on the NiTi microstructure and is highly related to the thermomechanical processing that is identified as proprietary by manufacturers. Whilst a detailed description of R-phase’s properties is beyond the scope of this manuscript, the reader is referred to a review article entitled ‘Physical metallurgy of Ti-Ni-based shape memory alloy’ by Otsuka & Ren (2005) as a good reference for detailed information on the structure and properties of R-phase NiTi.

Once the R-Phase was identified, wire in this state could not be ground, only twisted. This proprietary technology was used to optimize the molecular phase and properties of NiTi. This resulting crystalline structure modification, which has been shown to be finer than traditionally processed materials (Nicoll et al. 2006, summary data available at http://www.sybronendo.com/index/cms-filesystem-action?file=sybronendo-pdf/tf-brochure.pdf, accessed on 9/1/2011), maximized the instrument’s flexibility and resistance to breakage, with instruments exhibiting 36% more torque than instruments that were machined (Gambarini et al. 2008a, 2009, Testarelli et al. 2009a,b). The TF cutting flutes were created by twisting the file, which optimizes grain structure in the metal, as grinding is claimed to weaken the metal’s structure at the molecular level and create microfractures on the metal’s surface – both of which can lead to file fracture. A special conditioning treatment of the TF [proprietary process called Deox that removes the oxidation layer and any surface
impurities but does not remove any of the base material (Sabala 2010) finishes the surface of the file whilst respecting the integrity of the underlying grain structure. This process has been claimed to be the sole explanation for the improvement in torsional resistance and increase in instrument strength (Gambarini et al. 2008b, 2009). This outcome resulted in a rotary NiTi file that was strong enough to prepare the root canal in a safe manner whilst still being flexible enough to do it efficiently. In fact, because of the uniqueness and safety of this instrument, it has been claimed to be able to be used in multiple applied motions in the root canal system (Gambarini et al. 2010).

In a recently published assessment of the cyclic fatigue and fracture characteristics of the TF compared to ground files, Kim et al. (2010) examined the surface of the files with SEM prior to subjecting both file types to cyclic (rotational bending) fatigue testing. The TF showed a significantly higher resistance to cyclic fatigue than other NiTi files that were manufactured with a grinding process, although a parallel study showed that this finding is not universal relative to all ground files (Larsen et al. 2009). The path of crack propagation appeared to be different for TF-finished versus the non-surface-treated instruments. Whilst all specimens showed similar fractographic appearance, which indicated a similar fracture mechanism, instruments with abundant machining grooves (ground files) seemed to have a higher risk of fatigue. Most recently, however, multiple episodes of autoclave sterilization were shown to significantly decrease the cyclic fatigue resistance of the TF in a specific size (25/0.06) (Hilfer et al. 2011). Furthermore, when evaluating the TF with four other commercially available files for torsional resistance to fracture, the TF exhibited the lowest torsional resistance that was characterized by circular abrasion marks and skewed dimples near the centre of rotation at the point of fracture (Park et al. 2010).

Machined files

The majority of presently used NiTi files are machined. Because of some of the potential challenges encountered in this process and in the outcomes, based on clinical applications of these instruments, newer types of nitinol have been developed. Studies using atomic force microscopy graphically showed a significant difference in surface irregularities as a consequence of machining differences between various NiTi instrument manufacturers (Valois et al. 2005). The base of the surface irregularities acted as a stress inducer because an applied load would be concentrated at one point or area instead of being evenly distributed over a smooth surface. Data from an SEM study indicated that machining groove cracks containing dentinal debris that had been wedged into the surface defects resulted in instrument fracture (Alapati et al. 2005). Recommendations to address this problem were to use processes that minimized the production of surface cracks and to use NiTi alloys that improved fracture toughness.

At that time, the most commercially pure form of Nitinol was identified as SE or SE508 (Nitinol Devices and Components Inc., Fremont, CA, USA). The nickel component of the SE508 was 55.8 wt. % with titanium equalling the vast majority of the remainder with trace elements including O, C, Fe each at approximately 0.05 wt. %. This material was used for instruments and hardware in many branches of medicine, including orthopaedics for joint replacement and reconstruction, along with cardiovascular stents that alter their size upon introduction into the higher temperature of the human body (Johnson et al. 2008). For endodontic instruments, a casting is forged initially into a cylindrical shape prior to rotary swaging under pressure to create a drawn wire. The wire is then rolled to form a tapered shape with even pressure from a series of rollers applied to the wire. Additional processes include drawing the wire and annealing the wire, which is ultimately followed by drawing the actual profile or cross-sectional shape of the wire, imparting unique square, oblong or round shapes prior to machining into rotary instruments (Thompson 2000).

In the past 5 years, a replacement for the NiTi alloy used for endodontic instruments was developed (Berendt & Yang 2006). This variant alloy was composed of SE508 nitinol that had undergone a proprietary method of treatment, comprised of drawing the raw wire under specific tension and heat treatments at various temperatures, resulting in a material that includes some portion in both the martensitic and the premartensitic R-phase whilst maintaining a pseudoelastic state (Johnson et al. 2008). The new wire was named M-Wire (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA), and instruments made from this wire exhibited nearly 400% more resistance to cyclic fatigue than stock instruments of the same size whilst maintaining comparable torsional properties.

Subsequently, the M-Wire has undergone further and more extensive testing (Alapati et al. 2009a,b) to determine the origin of its improved mechanical properties and has been subjected to studies that have addressed its resistance to both cyclic and torsional
strengthening. Presence of Ti-Ni precipitates in both microstructures exhibiting pronounced evidence of alloy depending on the processing conditions and with the martensite for which the proportions of NiTi phases processing of M-Wire gave a microstructure containing X-ray energy-dispersive spectrometric analyses. The diffraction, and scanning electron microscopy with micro-X-ray energy-dispersive spectrometric analyses. The processing of M-Wire gave a microstructure containing martensite for which the proportions of NiTi phases depended on the processing conditions and with the microstructure exhibiting pronounced evidence of alloy strengthening. Presence of Ti$_2$Ni precipitates in both microstructures indicated that M-Wire and the conventional SE wire for rotary instruments are titanium-rich.

Larsen et al. (2009) focused on the ability of the newly developed NiTi instrument to resist cyclic fatigue when compared with traditionally ground NiTi rotary instruments. Amongst both .04- and 0.06-tapered files, size 20 M-Wire files performed significantly better than all other files tested with tip sizes of 25. Under these circumstances, the new manufacturing processes appeared to offer greater resistance to cyclic fatigue in a simulated canal model with different-sized files.

Al-Hadlaq et al. (2010) investigated cyclic flexural fatigue resistance of rotary files made from the first commercially available instrument made from M-Wire™ and compared them with two similar instruments made from a conventional NiTi rotary alloy – all at size 30, 0.04 taper. The results indicated that the M-Wire files had superior cyclic flexural fatigue resistance that was statistically significant when compared to the other two file types made from a conventional NiTi alloy. Within these parameters, the authors suggested that specifically sized NiTi rotary files made from the newly developed M-Wire alloy have better cyclic flexural fatigue resistance than files of similar design and size made from the conventional NiTi alloy.

Gao et al. (2010) compared the cyclic fatigue resistance of a new, most recently designed instrument made from M-Wire (Dentsply Tulsa Dental Specialties) and regular SE wire at two different rotational speeds. These tests were performed by rotating instruments in an artificially constructed stainless steel canal with 5 mm radius and 90° angle of curvature at two test frequencies: 300 and 500 rpm. The time to failure was recorded, and the total number of cycles to failure was calculated and compared for a total of 160 samples. More than 50% of broken files made of SE wire exhibited multiple crack-initiation sites compared with the single crack initiation on files made of M-Wire.

Findings to the contrary with M-Wire were identified by Gambarini et al. (2008b) when they compared instruments produced by using the twisted method, those using the M-Wire alloy and those produced by a traditional NiTi grinding process. Results indicated that the manufacturing process that produced NiTi rotary files that were twisted resulted in significantly more resistant to fatigue than those produced by the traditional machining process. Instruments produced with M-wire were not found to be more resistant to fatigue than instruments produced with the traditional machining process. An additional deviation in the outcomes for cyclic fatigue resistance with some specific sizes of M-Wire files was noted by Kramkowski & Bahcall (2009).

Based on present data, the advances in material processing appear to offer substantial benefits to the efficacy, efficiency, durability and safety of contemporary endodontic instruments. However, technological developments in metallurgy offer the possibility of further enhancement of these materials, with one of the most promising processes being post-machining heat treatment.

**Impact of heat treatment on NiTi materials**

Miura et al. (1986) claimed that they had developed a unique NiTi wire that exhibited unusual elasticity, which no other orthodontic wire had shown to that time. The wire delivered a constant force over an extended portion of the deactivation range, and this NiTi alloy wire was the least likely to undergo permanent deformation during activation. The new alloy exhibited a specific stress–strain curve unlike those of the other tested materials. Stress remained nearly constant despite the strain change within a specific range. This unique feature was considered as the manifestation of so-called superelasticity. Furthermore, heat treatment enabled the load magnitude at which superelasticity is reflected to be influenced and controlled by both temperature and time. Since then, thermal treatments have been shown to impact on some of the mechanical and SE properties and transformation characteristics of NiTi SMA (Hamanaka et al. 1989, Liu & McCormick 1994, Kuhn et al. 2001, Kuhn & Jordan 2002, Frick et al. 2005), depending on their thermomechanical history (Frick et al. 2005).
particular, superelasticity and shape memory are strongly affected by heat treatment (Yoneyama et al. 1993, 2002, Thompson 2000, Kuhn et al. 2001, Kuhn & Jordan 2002). Furthermore, when heat treatment is coupled with instrument configuration, the mechanical properties of the metal may be altered significantly, resulting in either favourable or unfavourable outcomes, although specific thermal manipulations have resulted in enhanced alloy flexibility (Hamanaka et al. 1989). Kuhn & Jordan (2002) recommended annealing temperature around 400 °C, which allowed for a compromise between an adequate density for the R-Phase germination and a low density for the brittleness of the NiTi instrument. This process would be carried out prior to the machining of the instrument to decrease the work hardening of the alloy (Kuhn et al. 2001).

Hayashi et al. (2007) investigated the bending properties of hybrid rotary NiTi endodontic instruments in relation to their transformation behaviour. Four types of NiTi rotary endodontic instruments with different cross-sectional shapes (triangular-based and rectangular-based) and different heat treatment conditions (SE type and hybrid type with SME) were selected to investigate bending properties and phase-transformation behaviour. The bending load values of the hybrid type that had undergone additional heat treatment at the tip were significantly lower than those of the SE type with no additional heat treatment. The bending load values of rectangular-based cross-sectional-shaped instruments were significantly lower than those of triangular-based cross-sectional-shaped instruments. Phase-transformation temperatures of the hybrid type were significantly higher \( (P < 0.05) \) than those of the SE type. They indicated that additional heat treatment of hybrid NiTi instruments may be effective in increasing the flexibility of NiTi rotary instruments.

Zinelis et al. (2007) attempted to determine the effect of various thermal treatments on the fatigue resistance of a NiTi engine-driven endodontic files. Fifteen groups of five files each of size 30 and 0.04 taper were tested in this study. The cutting tip (5 mm from the end) of files is in a wide range of temperatures from 250 to 550 °C. Files heat-treated within the 430 and 440 °C groups showed the highest values, with fatigue resistance decreasing for thermal treatment at lower and higher temperatures. The authors speculated that appropriate thermal treatment may significantly increase the fatigue resistance of the NiTi files tested.

Yahata et al. (2009) investigated the effect of heat treatment on the bending properties of NiTi (SE alloy) endodontic instruments in relation to their transformation behaviour. The heat treatment temperature was set at 440 or 500 °C for a period of 10 or 30 min. The transformation temperature was higher for each heat treatment condition compared with the control. Two clear thermal peaks were observed for the heat treatment at 440 °C. The specimen heated at 440 °C for 30 min exhibited the highest temperatures, with subsequently lower temperatures observed for specimens heated at 440 °C for 10 min, 500 °C for 30 min, 500 °C for 10 min and control specimens. The sample heated at 440 °C for 30 min had the lowest bending load values, both in the elastic range (0.5 mm deflection) and in the SE range (2.0 mm deflection). The influence of heat treatment time was less than that of heat treatment temperature. The authors determined that changes in the transformation behaviour by heat treatment are effective in increasing the flexibility of NiTi endodontic instruments. However, because the useful life span of NiTi rotary endodontic instrument is unpredictable and plastic deformation of these instruments is seldom observed during or after usage, vis a vis stainless steel instruments, the use of heat-induced or heat-altering manipulations may influence or alter the properties that once were cited as being the main reasons for pursuing the use of NiTi instruments in endodontics.

Proprietary thermomechanical processes that are used to develop future NiTi formulations will undoubtedly create complex microstructures that will require multiple bench-top and clinical analyses. However, in the long term, the use of heat treatment to control these new microstructures may provide a relatively inexpensive method for creating superior rotary NiTi endodontic instruments (Alapati et al. 2005). To some extent, this pursuit has already resulted in the initial development of unique instruments that either do not display memory or have a ‘controlled memory’ (HyFLEX® CM, controlled memory NiTi files; Colte`ne/Whaledent, Inc., Cuyahoga Falls, OH, USA), as opposed to what is found with standard SE forms of NiTi. Alterations in the metal characteristics of this new instrument have resulted in claims of resistance to fracture, increased flexibility and canal tracking or centring ability that are superior to other available instruments (product brochure – http://www.hyflexcm.com/DevDownloads/30464A_HYFLEX-CM_bro.pdf). In fact claims have been made that this instrument is more than 300% more resistant to cyclic fatigue than standard NiTi files.

A second development in this area has been the heat treatment of TF. In recent studies, the post-twisting
phase-transformation temperatures of TF were significantly higher than those of machined files, with the bending load values being significantly lower for these files in the elastic and SE ranges (Hou et al. 2011). The application of this heating process has also been applied to machined files with the aim to transform the alloy into a slightly different crystalline phase structure, having enhanced mechanical properties (improved flexibility with superior mechanical resistance) (Gambarini et al. 2011). These achievements would allow for the shaping of root curved root canals with minimal risk of canal transportation, reduced instrument fracture and a reduction in adverse alterations in the root dentin.

In many respects, the outcome of post-machining or post-twisting heat treatment is diametrically opposed to what clinicians have favoured about NiTi instruments since their inception, that being the SE shape recovery. However, in an effort to understand the nature of the metal alterations and their impact on enhancing resistance to cyclic fatigue in particular, an international standard for cyclic fatigue testing of NiTi rotary instruments should be required to ensure uniformity of methodologies, so that resulting data can be compared (Plotino et al. 2009, Testarelli et al. 2009a,b).

Conclusion

Endodontic instruments made of NiTi shape-memory alloy have had a revolutionary impact on root canal treatment. The unique material properties of NiTi make it particularly suited for endodontic rotary instruments. This development has fostered a significant amount of research that is focused on enhancing metallic properties to improve clinical performance and safety because property and performance of endodontic files can significantly affect the outcome of root canal shaping and cleaning. Rotary NiTi instruments with improved reliability, durability and performance have evolved from this research with breakthroughs in file design and cost-effective fabrication. The integration of surface engineering (implantation or electropolishing) and/or microstructure control (heat treatment or innovative manufacturing techniques) into the endodontic file design has resulted in more favourable outcomes for instrument flexibility, fatigue resistance and cutting efficiency.

References


