Characterization of New Rotary Endodontic Instruments
Fabricated from Special Thermomechanically Processed NiTi Wire

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ABSTRACT

Although NiTi rotary instruments are very popular for endodontic treatment, instrument separation is still a challenge in clinic. A new NiTi rotary instrument (GT® Series X™, Dentsply Tulsa Dental Specialties) has recently been marketed that is machined from a wire (termed M-Wire) that has been subjected to a proprietary novel thermomechanical processing procedure. The manufacturer has claimed that this new M-Wire instrument has considerably improved flexibility and resistance to cyclic fatigue, compared to conventional rotary instruments that are machined from superelastic (SE) austenitic NiTi wire. Clinical use has confirmed that these new GT® Series X™ rotary instruments have outstanding clinical fatigue resistance (private communication from Dr. John Nusstein, Division of Endodontics, College of Dentistry, The Ohio State University). However, the mechanism for the improved clinical performance of these instruments is unknown.

The objective of this study was to employ a variety of metallurgical laboratory techniques to determine the origin of these improved mechanical properties for the new rotary instruments. Specimens from as-received M-Wire instruments, clinically used M-Wire instruments, and conventional instruments made from SE wire were prepared for evaluation. The temperature range for
phase transformation was examined by differential scanning calorimetry (DSC). Vickers hardness measurements were made since hardness variations for the same type of alloy has been found to correlate with variations in mechanical properties. The microstructures of the NiTi alloys were revealed by acid etching and examined with an optical microscope and a scanning electron microscope that was also capable of X-ray energy-dispersive spectrometric analyses (SEM/EDS). Wear resistance of clinically used M-Wire instruments was investigated by examining their surfaces with an SEM. In a complementary study, bright-field images of M-Wire blanks were obtained by scanning transmission electron microscopy (STEM). STEM images from that study are included in this dissertation to provide further insight into the mechanism for the mechanical properties of the M-Wire instruments.

DSC study showed that M-Wire instruments have much higher $A_f$ (austenite-finish) temperatures (over $40^\circ$C) than conventional superelastic rotary instruments (below room temperature), and are a mixture of martensite, R-phase and austenite at room temperature. The Vickers hardness of M-Wire instruments is significantly higher than that of conventional rotary NiTi instruments. Mean Vickers hardness number for the tip, intermediate region and shank region of size 30/.04 taper M-Wire instruments were about 374, 380 and 392 each, whereas values of Vickers hardness number for the corresponding regions of conventional instruments were typically less than about 320, 350 and 380.

Better wear resistance was observed with the SEM on clinically used M-Wire instruments, which presented less microcracks and evidence of
permanent deformation on the surface compared with surfaces of clinically used conventional NiTi instruments. This improved wear resistance is attributed to increased hardness for surface region of the M-Wire instrument. Acid-etched M-Wire instruments (surfaces and cross-sections) presented a classical lenticular martensite structure when observed with the optical microscope and SEM. EDS analyses of the microstructures of the M-Wire instruments revealed titanium-rich precipitates. The complementary STEM examinations of M-Wire blanks revealed much coarser grains, twinning, and a high density of dislocations, which were not observed in starting superelastic NiTi wire blanks for conventional instruments.

In summary, increased hardness, which is indicative of higher strength and improved wear resistance, was found for M-Wire instruments, compared with conventional superelastic ProFile® instruments, which served as a control for this study. The STEM observations show that the improved mechanical properties of the starting M-Wire (and the rotary instruments manufactured from this special NiTi wire) arise from strengthening mechanisms in the martensitic structure, which were induced by extensive thermomechanical processing.
DEDICATION

This dissertation is dedicated to my family, parents and friends who have supported me.
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CHAPTER 1

INTRODUCTION

1.1 Nickel-titanium instruments in clinical use and metallurgy of NiTi alloys

1.1.1 Nickel-titanium instruments in clinical use

Conventional endodontic treatment involves removal of inflamed or necrotic pulp tissue and filling of the canal with therapeutic materials (Cohen et al, 2002). The morphology of the root canal system and the curvature of the roots vary (Manning, 1990a and 1990b; Cohen and Burns, 2002), and these factors provide great challenges for dentists. Because of the lower elastic modulus and wide elastic working range of the nickel-titanium (NiTi) alloy compared with stainless steel (Walia et al, 1988), nickel-titanium instruments were introduced into endodontic treatment in 1990's; with the first rotary instrument (ProFile®, Tulsa Dental) marketed in 1993.

Nickel-titanium rotary instruments have thus been available in endodontics for over 15 years. Compared with root canal instruments made from stainless steel, these nickel-titanium instruments have better flexibility, which is one of the
most important requirements for rotary instruments to negotiate curved root canals (Tepel et al, 1997). Flexibility allows appropriate canal enlargement while maintaining the instrument centered within the canal to preserve tooth structure and prevent transportation (Tepel et al, 1997). Although rotary instrument made from NiTi alloy are quite flexible compared with stainless steel instruments when values of elastic modulus are considered, the instrument flexibility is also inversely related to the cross-section dimensions. Therefore the rigidity of an instrument (inverse of elastic flexibility) increases with larger size and taper. Although manufacturers have made some changes in instrument design in order to improve the flexibility, canal transportation still occurs, due to the tendency of NiTi instruments to straighten when the elastic limit of the NiTi instruments is exceeded in a very curved root canal (Pruett et al, 1997). This suggests that NiTi rotary instruments with improved mechanical properties are required (Iqbal et al, 2007).

Fracture and separation of NiTi rotary instrument during clinical use are also a problem (Patino et al, 2005). Due to its lower yield strength and ultimate tensile strength compared with stainless steel (Tables 1.1 and 1.2), NiTi endodontic instruments are more susceptible to fracture at lower loads than for stainless steel instruments (Anusavice, 1991). Martin et al (2003) reported that unexpected fractures might occur without any visible changes in the NiTi instruments, such as an evident defect or permanent deformation. Based on the examination of clinically used NiTi instruments, Sattapan et al (2000) reported a fracture frequency of 21% from 378 discarded Quantec instruments, but a much
lower fracture frequency of about 5% was subsequently reported in another study (Parashos et al, 2004). Failure analysis by Alapati et al (2005) has shown that the fracture pattern is complex, and consists of both brittle and ductile aspects. Attempts to remove separated instrument fragments could result in excessive removal of dentin, ledging, perforation, and even extrusion of the fractured part out of apex (Fors and Burg, 1986; Hülsmann, 1993). However, not removing the file could result in leaving behind necrotic pulp tissue, if the instrument separation occurs at the early stage of canal preparation. Therefore the prognosis for successful root canal treatment would be greatly reduced by NiTi rotary instrument separation during instrumentation (Torabinejad et al, 2002; Madarati and Watts, 2008).

Therefore, for an ideal NiTi endodontic instrument, the ultimate tensile strength should be high enough to resist separation, and the instrument should have high flexibility to avoid canal perforation and allow high resistance to fatigue (Thompson, 2000). Also, the cutting ability should be high enough to prepare the root canals efficiently.

1.1.2 Metallurgy and phase transformations of NiTi alloys

The nickel–titanium alloy was first developed in the 1960s. This alloy was named Nitinol, an acronym for the elements from which the material was composed (Ni for nickel and ti for titanium) and the location for these investigations (nol from the Naval Ordnance Laboratory). Based on the equiatomic, intermetallic compound NiTi, the alloy composition used for the
manufacture of NiTi instruments is about 55% nickel and 45% titanium (wt.%) (Thompson, 2000). A review article (Thompson, 2000) discusses the properties of this nickel-titanium alloy and the general manufacturing process for the NiTi rotary endodontic instruments, which are used with a slow-speed dental handpiece. These instruments are machined from NiTi wire blanks, in contrast to the manufacturing process used for the stainless steel files and reamers, which are manufactured by twisting tapered wire blanks. The properties of the NiTi and stainless steel alloys are summarized in Tables 1.1 and 1.2, as previously noted. Thompson (2000) also discussed the role of the Ni:Ti ratio, incorporation of other trace elements in the alloy composition, and heat treatment on the properties of the NiTi alloys.

There are three major forms for nickel-titanium (NiTi) alloys for orthodontic use: superelastic, nonsuperelastic, and shape memory (Bradley et al, 1996; Brantley, 2001). The mechanical behavior of these three different forms of NiTi alloys arises from their microstructural phases and the character of these phases (Brantley, 2001). There are three major phases in these NiTi alloys. The austenite phase has a complex body-centered cubic structure, and exists at higher temperatures and lower stresses. In contrast, the martensite phase exists at lower temperatures and higher stresses, and has a monoclinic crystal structure. Transformation between these two phases occurs by twinning, which is reversible. The R-phase is an intermediate phase that can form during the forward and reverse transformation between austenite and martensite phases. Due to the narrow range of the equiatomic NiTi phase field in the nickel-titanium
phase diagram (Goldstein et al., 1987), shown in Figure 1.1, Ti$_2$Ni and Ni$_3$Ti precipitates can form in Ti-rich and Ni-rich alloys, respectively, during cooling from elevated temperatures. Thompson (2000) has suggested that oxide particles form during the manufacturing process of the NiTi alloys, which react with the ambient oxygen. The presence of such oxide particles in rotary nickel-titanium instruments has been reported by Alapati et al. (2003), as shown in Figure 1.2.

The phase transformation temperatures that govern the mechanical properties of NiTi alloys are as follows: martensite-start temperature ($M_s$), martensite-finish temperature ($M_f$), austenite-start temperature ($A_s$) and austenite-finish temperature ($A_f$), along with the corresponding transformation temperatures for the start ($R_s$) and finish ($R_f$) of the formation of the intermediate R–phase (Brantley, 2001; Brantley et al., 2002a, 2002b and 2002c).

1.2 NiTi rotary instrument fracture and related factors

1.2.1 Metallurgy and fracture

The reasons for fracture of rotary NiTi instruments are complex, and understanding the mechanisms for failure could provide insight for instrument design and the manufacturing process. As previously noted, compared with the manufacture of stainless steel instruments, the manufacture of NiTi endodontic instruments is more complex because these files (until the recently marketed Twisted Files from SybronEndo) have to be machined from wire blanks rather
than twisting the wire blanks (Thompson, 2000). Manufacturing NiTi rotary instruments by twisting the wire blanks would likely result in instrument fracture (Schäfer, 1997), except for the new Twisted Files.

Surface imperfections such as scratches, transitional angles, microcavities, and debris, are introduced during the manufacturing process (Walia et al, 1988; Eggert et al, 1999; Kuhn et al, 2001). Chianello et al (2008) found that no NiTi instrument was free of imperfections and most presented 2 to 7 types of surface defects. The relationship between manufacturing imperfections and breakage of rotary instruments has been investigated (Kuhn et al, 2001; Cheung et al, 2007a). Surface imperfections may serve as stress concentrators and induce crack initiation and propagation, resulting in reduced fatigue life (Alapati et al, 2003; Borgula et al, 2005; Cheung et al, 2007a). However, a study by Cheung et al (2007b) showed that surface smoothness from electropolishing did not enhance the low-cycle fatigue resistance of rotary instruments. So the effect of instrument surface defects on fatigue failure is still controversial. The role of dentin chips during crack propagation has been proposed (Alapati et al, 2004), but other studies have suggested that the dentin may be adhering to surface regions containing carbon and sulfide and are not fully removed during sample preparation (Martins et al, 2002; Parashos et al, 2004a).

There are two classifications of metal fracture: ductile and brittle. Ductile fracture means the metal undergoes plastic deformation before it breaks, but there is little or no plastic deformation in brittle fracture (Askeland et al, 2003). Typically, there is a crack initiation site at the metal surface, and propagation of
the crack will occur because of stress concentration at the crack tip. Examination of a fracture surface using the scanning electron microscope (SEM) provides useful information to characterize the major aspects of the failure processes (LeMay, 1978; Alapati et al, 2005; Borgula et al, 2005). In ductile fracture, microvoids are formed in the metal, and coalescence of the microvoids finally weakens the material, leading to fracture. In brittle fracture, cracks spread along different planes and radiate from the initiation site (Askeland et al, 2003).

Ductile fracture is characterized by a dimpled surface appearance, as shown in Figure 1.3, and was generally identified in clinically separated NiTi rotary instruments (Alapati et al, 2005). The nucleation of secondary phase particles in the microstructure, such as nickel-titanium oxides (Duerig et al, 1990), was suggested as the main reason for the dimpled surface appearance. A more complex fracture surface was observed in clinically retrieved ProFile® GT® instruments, arising from transgranular fracture across the fine grains and intergranular fracture (Alapati et al, 2005). Two other major fracture processes observed for the clinically retrieved NiTi instruments were torsional deformation without separation and axial fracture (Alapati et al, 2005).

The fracture of NiTi rotary instruments during clinical use may due to cyclic loading or a single episode of sudden overload. Previously it had been assumed that the fracture and separation of NiTi endodontic instruments were mainly due to cyclic loading (Parashos et al, 1994). However, recent studies suggest that fracture can be due to sudden overload rather than cyclic loading, based on the observation of absence of characteristic striations at the fracture
surface that are associated with fatigue fracture (Alapati et al, 2005; Spanali-Voreadi et al, 2006).

Work hardening, which occurs from plastic deformation below the recrystallization temperature (at which the cold-worked microstructure is replaced by new stress-free grains), strengthens a metal by the formation of a high density of dislocations (Engineer Edge). But studies have suggested that work hardening of NiTi rotary instruments induced during the manufacturing process and clinical use may be detrimental to their mechanical properties (Kuhn et al, 2002; Alapati, 2006; Parashos et al, 2006). Work hardening at the tip region of the files due to the manufacturing process, also may have a role for fracture of NiTi endodontic instruments (Kuhn et al, 2002; Alapati, 2006). Rotating NiTi instruments in curved root canals are subjected to fluctuating tensile and compressive stresses, which may result in work hardening of the metal and induce the initiation of microcracks (Parashos et al, 2006).

1.2.2 Other factors contributing to the separation of NiTi rotary instruments

Other factors have been linked to the fracture (often termed separation in endodontics) of NiTi rotary instruments during clinical use. These factors include operator skills, preparation techniques, anatomy of the root canal system, number of instrument uses, and dimensions of the instruments. Chemical and heat sterilization agents may also affect the mechanical properties of rotary instruments.
Operator skills and preparation techniques

Clinical studies have suggested that operator is the most important influence for the failure of NiTi rotary instruments (Regan et al, 2000; Parashos et al, 2004a). Sufficient clinical training and adequate instrumentation skills are essential for practitioners. Also, manufacturer guidelines to prevent instrument fracture should be followed (Di Fiore, 2007). Varying instrumentation sequences and use of combinations of different file tapers have been suggested to prevent torsional and fatigue failure of the NiTi rotary instruments (Schrader et al, 2005). Also, preflaring of the root canal with hand instruments before use of a rotary NiTi instrument has been highly recommended (Tan et al, 2002; Roland et al, 2002; Varela et al, 2005). In addition, the use of lubricant is a must, due to its effect of reducing the friction and the number of resulting surface defects for NiTi rotary instruments during instrumentation (Parashos et al, 2006).

Root canal anatomy

Root canal anatomy can be very complicated. Although an X-ray is taken before root canal treatment, the three-dimensional structure of a complex root canal system normally cannot be fully appreciated from information provided by a two-dimensional picture. The angle and radius of canal curvature are two parameters normally used to evaluate the morphology of the canal system (Gunday et al, 2005). It has been accepted that the more complicated the root canal morphology, the higher is the chance of instrument fracture. Most
Instrument fracture has been reported for treatment of molars, especially at the apical third of the canal (Peng et al, 2005; Iqbal et al, 2006). The fatigue rate of instruments undergoes an increase with the decreased radius of curvature and increased angle of curvature of the root canal (Li et al, 2002; Zelada et al, 2002). Compared with the angle of root canal curvature, the radius of canal curvature appears to be an important factor in instrument fracture (Madarati et al, 2008).

**Number of clinical uses**

There are different opinions about how many times rotary NiTi instruments should be used in the clinic. The manufacturer suggests one time of clinical use, and similar suggestions were proposed by one study (Arens et al, 2003) to reduce fracture frequency, but other studies have reported that NiTi rotary instruments may be used up to ten times in simulated canals, or to prepare 4 molar teeth without fracture (Yared et al, 1999; Peters et al, 2002). However, it is generally accepted that prolonged clinical use of rotary NiTi instruments significantly reduces their cyclic flexural fatigue resistance (Yared, et al, 2004; Fife et al, 2004; Bahia et al, 2005). Since individual root canals are not anatomically the same, as well as other factors, it is difficult to give general recommendations on the appropriate number of uses for NiTi rotary files.

**Instrument design**

Cross-section shape and size are the two most important factors in rotary instrument design that determine their fatigue resistance during clinical use.
Compared with a U-shaped cross-section, a triangular-shaped cross-section for rotary instruments is reported to be stronger and more resistant to bending forces. This could be due to more even and favorable force distribution (Berutti et al, 2003; Schäfer et al, 2003). However, U-flute designed instruments are more flexible than triangular triple-helix instruments (Turpin et al, 2000; Berutti et al, 2003). A study by Berutti et al (2003) provided suggestions on the clinical use of triangular-shaped ProTaper® and U-shaped ProFile® instruments. The ProTaper® file is more appropriate in the initial canal-shaping phase and should be used in narrow and curved canals. However, the ProFile® is more appropriate in the final stage of shaping and should be used in wide and curved canals (Berutti et al, 2003).

Taper of NiTi instruments was found to be important in determining the time for fracture. The time to instrument fracture decreased with an increase of taper (Haikel et al, 1999). Also, instrument size (diameter) has a strong influence on performance (Chaves Craveiro de Melo et al, 2002). The tensile stress on the external surface of the instrument with a larger diameter is higher, compared with a smaller diameter instrument. Moreover, a larger size instrument has a greater area to contact the dentin walls; therefore it will experience greater friction and more internal stress accumulation (Haïkel et al 1999; Ullmann et al, 2005). However, the increased size also means improved strength due to a larger cross-section area (Yared et al, 2003; Guilford et al, 2005).

Radial lands are the flat regions on the cutting surfaces and have an important role in determining the strength of the files. The less the extent of the
radial lands, the less is the resistance of the instrument to torsional stress (Gambarini, 2005). The K3 rotary instruments (Sybron Endo, Orange, CA) are characterized by wide radial lands, radial land relief and a slightly positive rake angle. Radial land relief reduces friction on the canal wall, and the positive rake angle provides the active cutting action of the K3 file (Gambarini, 2005). However, one study has shown that K3 files were significantly less flexible than other NiTi rotary instruments with the same size and taper, and this may be due to the bulk cross-section design (Schafer et al, 2003).

**Cleaning agent and heat sterilization**

Sodium hypochlorite, a material used for sterilization and lubrication during root canal preparation, is very corrosive to NiTi alloys (Lasley et al, 2004). Studies have shown that NiTi instruments are susceptible to corrosive attack by sodium hypochlorite with concentration varying from 1.2% to 5.25%, and that the resulting corrosion pits formed on instrument surfaces appear to be detrimental to low-cycle fatigue life (Busslinger et al, 1998; Cheung et al, 2008). The concentration of sodium hypochlorite seems to be relevant to corrosion effects on NiTi rotary instruments (Busslinger et al, 1998). Their study suggested that statistically significant corrosion occurred after 30 minutes immersion of LightSpeed® instruments in 5% sodium hypochlorite, compared with 1% sodium hypochlorite. However, other studies suggested that the bulk mechanical properties of rotary instruments were not affected by immersion in sodium hypochlorite (Hakel et al, 1998; O'Hoy et al, 2003; Darabara et al, 2004).
Therefore, the effect of sodium hypochlorite on the clinical performance of NiTi rotary instruments is still controversial.

Heat sterilization of new or used rotary instruments exposes them into a repeated heating/cooling cycle. Several studies have focused on the influence of heat sterilization on the fatigue resistance of NiTi rotary instruments, but currently the results are contradictory and there is no consensus (Serene et al, 1995; Silvaggio et al, 1997; Canalda-Sahli et al, 1998; Mize et al, 1998; Svec et al, 1999; Hilt et al, 2000; Chaves Craveiro de Melo et al, 2002). The study by Canalda-Sahli (1998) suggested that generally there was slight decrease in the flexibility of NiTi files after 10 cycles of heat sterilization, but all files tested satisfied the minimum ISO requirements for angular deflection after sterilization. However, Serene et al (1995) reported that sterilization increased the fatigue life of rotary instruments through the increase in hardness and torsional resistance of the NiTi alloy. Similar results were found in the study by Chaves Craveiro de Melo et al (2002). After five cycles of heat sterilization, the fatigue resistance of ProFile® instruments to cyclic rotation in simulated root canals and their microhardness were significantly increased.

1.3 Methods to improve fatigue resistance of NiTi endodontic instruments

Several strategies have been employed to improve the fatigue resistance of NiTi endodontic instruments. These strategies include electropolishing, ion implantation, surface coatings and heat treatment. Ion implantation of NiTi instruments was first introduced by Lee et al (1996), and proved to be an
effective method to increase surface hardness and wear resistance, resulting in better cutting efficiency of the rotary instruments. Thermal nitridation and nitrogen-ion implantation treatment of NiTi files were applied to yield a higher N:Ti ratio and increased cutting ability (Rapisarda et al, 2000). Physical vapor deposition was employed by Schäfer (2002) to increase the fatigue resistance of NiTi instruments, and the cutting efficiency of surface-coated NiTi files was increased by up to 26.2% compared to uncoated instruments. Although these surface treatments have obvious advantages for improving fatigue resistance and cutting efficiency of NiTi rotary instruments, their high costs limit wide usage among manufacturers.

Electropolishing has been used by some manufacturers to improve the surface finish (Lausmaa et al, 2001). Some studies have reported that it is an effective way to increase instrument fatigue resistance by reducing the presence of microcracks and machining damage, which was previously suggested to play a role in instrument stress concentration and crack propagation (Walia et al, 1988; Eggert et al, 1999; Kuhn et al, 2001; Tripi et al, 2006). However, another study found that the low fatigue life of rotary instruments was not affected by the surface electropolishing procedure (Cheung et al, 2007). Similar results were found in a study by Barbosa et al (2008); there was no effect of electrochemical polishing on the fracture resistance of K3 (SybronEndo) rotary instruments.

The clinically relevant properties of NiTi biomedical alloys depend on the thermomechanical processing history used by the manufacturer (Johnson, 2009). Previous studies of NiTi orthodontic wires have shown that the effect of heat
treatment depends on both temperature range and heating time (Miura et al, 1986; Khier et al, 1991). For example, the bending properties of superelastic NiTi orthodontic wires are not affected by heat treatment at 400°C, whereas the superelastic behavior would be lost after heat treatment at 600°C. For heat treatment at 500°C, the average superelastic bending moment for NiTi orthodontic wires would be decreased by prolonged heat treatment (for example, 2 hours), compared with minimal effect on the cantilever bending plots for a heat treatment time of 10 minutes (Miura et al, 1986; Khier et al, 1991).

The effect of heat treatment on the mechanical properties of NiTi rotary instruments was examined by Kuhn and colleagues (2001 and 2002). Annealing at temperatures around 400°C was found to yield a superior microstructure, resulting in increased instrument flexibility and lower brittleness. However, increased brittleness of instruments was observed after the annealing temperature was increased higher than 600°C (Kuhn et al, 2001 and 2002). Similar results were reported by Zinelis et al (2007), who found that the fatigue resistance of a commercial rotary NiTi file was steadily increased from the as-received state with increasing heat treatment temperature to 440°C, and then decreased with further increase in the heat treatment temperature to 550°C. These latter investigators suggested that heat treatment at temperatures higher than 600°C would cause recrystallization of the microstructure, which should be avoided for rotary instruments (Kuhn et al, 2002; Zinelis et al, 2007). Interestingly, decreased surface hardness of the heat-treated instruments was observed, which was attributed to elimination of work hardening (Kuhn et al, 2001 and 2002;
Zinelis et al, 2007). In general, thermomechanical processing seems to be a very promising way to increase the fatigue resistance of NiTi endodontic files. Alapati reported (2006) the results from an extensive study of the effects of heat treatment on phase transformations in NiTi rotary instruments. He found that heat treatments at 400°C, 500°C and 600°C raised the $A_t$ temperature of ProFile® GT® to 45°C – 50°C, however, heat treatment at 850°C caused drastic changes in transformation behavior: the DSC curves were very complex with irregular peaks. He suggested that high temperature induced the change in NiTi file microstructure. Similar results were found in studies by other investigators, who suggested that heat treatment at temperatures higher than 600°C would cause recrystallization of the microstructure, which should be avoided for rotary instruments (Kuhn et al, 2002; Zinelis et al, 2007).

1.4 M-Wire rotary endodontic instruments and significance of research

Currently, a new type of nickel-titanium (NiTi) wire (named M-Wire) has been processed by an extensive thermomechanical procedure (Sportswire LLC), and this wire is reported to have superior laboratory fatigue performance compared to conventional NiTi superelastic (SE) wire used for the manufacture of rotary instruments (William Ben Johnson, private communication). This new M-Wire has greatly enhanced fracture resistance compared with conventional superelastic wire, along with a higher ratio of tensile strength to upper superelastic plateau stress (William Ben Johnson, private communication), as shown in Figure 1.4. Our research group has investigated the microstructures of
M-Wire by scanning transmission electron microscopy (STEM), temperature-modulated differential scanning calorimetry (TMDSC), Micro-X-ray diffraction, and scanning electric microscopy (Brantley et al, 2008; Alapati et al, 2008; Iijima et al, 2008; Buie et al, 2008), and obtained information about the phase transformations in M-Wire. The scanning transmission electron microscopy study (Brantley et al, 2008) found presence of martensite and perhaps R-phase in the cross-sections of M-Wire, which is absent in the microstructure of conventional SE wire. Micro-X-ray diffraction analyses at room temperature suggest that M-Wire is a mixture of austenite, martensite and R–phase (Figure 1.5). This result is complementary to the temperature-modulated differential scanning calorimetry (TMDSC) analysis by Alapati et al (2008). They found that the austenite-finish temperature of M-Wire (45°C – 50°C) is much higher than that for conventional superelastic wire (approximately 20°C or lower), as shown in Figure 1.6. The microstructural study by Buie et al (2008) found the classic lenticular appearance of martensite in the microstructure of M-Wire (Figure 1.7). Energy-dispersive spectrometric (EDS) analyses with the SEM have shown that the precipitates in M-Wire are Ti$_2$Ni, indicating that this alloy is Ti-rich. Studies by our group have found that the thermomechanical processing for M-Wire yields a different microstructure and phase transformation temperature range for this new M-Wire for rotary instruments compared to conventional SE wire.

Rotary instruments made from M-Wire have been recently introduced to the market (GT$®$ Series X$™$, Dentsply Tulsa Dental Specialties). This manufacturer claims that the new M-Wire instruments have increased cyclic
fatigue resistance and flexibility. Extensive clinical use in the Graduate Endodontic Clinic of the College of Dentistry has confirmed the superior cyclic fatigue and fracture resistance of these instruments (Dr. John Nusstein, private communication). A recent study by Watanabe et al (2009) found that the cyclic fatigue resistance of GT® Series X™ instruments was significantly better than the fatigue resistance for another new generation of NiTi rotary instruments [Twisted File (TF) (SybronEndo)] and for the conventional NiTi rotary instruments evaluated [EndoSequence™ (Brasseler USA) and ProFile® (Dentsply Tulsa Dental)].

However, there are no published studies on the mechanism for the improved clinical performance of M-Wire instruments. This study was designed to investigate the microstructure and phase transformations for M-Wire rotary instruments in both the as-received and clinically used conditions, for comparison with previously published information about conventional NiTi rotary instruments. The goal of this research project was to understand the mechanism of the increased cutting efficiency and fatigue resistance of M-Wire rotary instruments. The ultimate goal of our research group is to provide new insight about the rotary instruments made from M-Wire, which will lead to further improvement in their clinical performance.

1.5 Hypotheses and specific aims

The hypotheses for this research project are that (a) the thermomechanical processing used for M-Wire that is machined into rotary
endodontic instruments results in significant changes in the microstructure and phase transformation behavior, compared to conventional rotary instruments, and that (b) the altered microstructure and phase transformation behavior is beneficial to the improved clinical performance of these new rotary instruments.

The specific aims of this research project are:

(1) To study the effect of thermomechanical processing on the phase transformations of as-received and clinically used M-Wire instruments;

(2) To study the effect of thermomechanical processing on the microstructure of new and clinically used instruments;

(3) To study the effect of thermomechanical processing on the hardness of M-Wire instruments, and;

(4) To study the effect of thermomechanical processing on the wear resistance of clinically used instruments.
<table>
<thead>
<tr>
<th>Property</th>
<th>55-Nitinol Austenite</th>
<th>55-Nitinol Martensite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm(^3))</td>
<td>6.45</td>
<td></td>
</tr>
<tr>
<td>Coefficient of thermal expansion (°C(^{-1}))</td>
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<td></td>
</tr>
<tr>
<td>Young's modulus (GPa)</td>
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<td>50</td>
</tr>
<tr>
<td>Yield strength (MPa)</td>
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</tr>
<tr>
<td>Ultimate tensile strength (MPa)</td>
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<td></td>
</tr>
<tr>
<td>Elongation</td>
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<td></td>
</tr>
<tr>
<td>Shape memory transformation temperature (°C)</td>
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</tr>
<tr>
<td>Latent heat of Transformation (J/g)</td>
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<td></td>
</tr>
<tr>
<td>Shape memory recoverable strain</td>
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</tr>
<tr>
<td>Super-elastic Recoverable strain</td>
<td>Up to 8%</td>
<td></td>
</tr>
<tr>
<td>Transformation fatigue life at 6% strain</td>
<td>Several hundred cycles</td>
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</tr>
<tr>
<td>Transformation fatigue life at 2% strain</td>
<td>(10^5) cycles</td>
<td></td>
</tr>
<tr>
<td>Transformation fatigue life at 0.5% strain</td>
<td>(10^7) cycles</td>
<td></td>
</tr>
</tbody>
</table>

Table 1.1 Typical properties of nitinol alloys (Thompson, 2000).
<table>
<thead>
<tr>
<th>Property</th>
<th>Austenitic Stainless Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>17-20% Cr, 8-12% Ni, 0.15% C, balance mainly Fe</td>
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<tr>
<td>Modulus of elasticity (GPa)</td>
<td>160-180</td>
</tr>
<tr>
<td>Springback (As-received cond)</td>
<td>0.0060-0.0094</td>
</tr>
<tr>
<td>Springback (Heat-treated</td>
<td>0.0065-0.0099</td>
</tr>
<tr>
<td>condition)</td>
<td></td>
</tr>
<tr>
<td>Yield strength (MPa)</td>
<td>1100-1500</td>
</tr>
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</table>

Table 1.2 Properties of austenitic stainless steel alloys (Brantley, 2001)
Figure 1.1 Nickel-titanium binary phase diagram, showing region near intermetallic compound NiTi (Brantley, 2001 and originally from Goldstein et al, 1987).
Figure 1.2 SEM photograph of the cutting tip for a LightSpeed™ instrument after one simulated clinical use, showing elongated nickel-titanium oxide precipitates and flattening of the rollover (Alapati et al, 2003). Scale bar length is 5 μm.
Figure 1.3 Secondary electron image of the fracture surface of a Pro-Taper™ rotary instrument, showing elongated dimples indicative of ductile fracture and secondary phase particles which may be nickel-titanium oxides (original magnification, ×2500; scale bar length, 6 μm) (From Alapati et al, 2005).
Figure 1.4 Stress-strain plots for tensile testing of M-Wire (right) and conventional superelastic NiTi wire (left) (Provided by William Ben Johnson, Sportswire LLC).
Figure 1.5 Micro-X-ray diffraction patterns from segment of M-Wire (Sportswire LLC) at 25°C, showing the mixture of austenite, martensite and R–phase (Iijima et al, 2008).
Figure 1.6 DSC analyses for a test specimen of M-Wire segments (Sportswire LLC), showing higher austenite-finish temperature of M-Wire, compared to traditional superelastic NiTi wire for rotary instruments (Alapati et al, 2008).
Figure 1.7 Secondary electron SEM images (Buie et al, 2008) of conventional superelastic NiTi wire (left) and M-Wire (right).
CHAPTER 2

DIFFERENTIAL SCANNING CALORIMETRIC STUDIES OF THE PHASE TRANSFORMATIONS IN M-WIRE INSTRUMENTS

Abstract

Differential scanning calorimetric (DSC) analyses were performed between -80° and 150°C on single-segment (tip, middle and shank regions) specimens obtained from representative M-Wire instruments (GT® Series X™, Dentsply Tulsa Dental) in the as-received condition and after seven to eight times clinical use. The DSC analyses showed that each segment of the as-received or clinically used M-Wire instruments had an \( A_f \) (austenite transformation completion or austenite-finish) temperature exceeding about 40°C and that there were only small differences in \( A_f \) temperature for segments from the three regions of both the as-received and clinically used M-Wire instruments. Previous DSC studies showed that ProFile® and Lightspeed™ conventional NiTi rotary instruments have \( A_f \) temperatures substantially below mouth temperature. One endothermic peak was observed in most M-Wire instruments during the heating cycle, corresponding to transformation from martensite to R-phase, and then to
austenite. A single broad exothermic peak for the reverse transformation from austenite to martensite was generally observed during the cooling cycle. The higher $A_f$ temperatures of GT® Series X™ instruments is consistent with their martensite structure, which is observed at room temperature with the STEM, SEM and optical microscope in complimentary studies. Low values of less than approximately 3 J/g for the enthalpy change ($\Delta H$) for the transformation from martensite to austenite indicate that the M-Wire instruments predominantly contain stable martensite that does not undergo transformation.

2.1 Introduction

Nickel-titanium rotary instruments have become widely used in clinical practice after Walia et al (1988) introduced this alloy to the endodontic profession. The manufacture of nickel-titanium instruments for endodontics has been discussed in a review article (Thompson, 2000). The nickel-titanium alloys for endodontic instruments are based upon the equiatomic intermetallic compound NiTi (Brantley et al, 2001), and these alloys are similar to nickel-titanium alloys originally used for orthodontics (Andreasen et al, 1979).

Due to its lower elastic modulus and higher flexibility compared with stainless steel (Brantley et al, 2001), nickel-titanium rotary instruments are able to negotiate curved root canals (Walia et al, 1988; Thompson et al, 1997 and 1998; Bryant et al, 1999). Studies have shown that NiTi alloys used to manufacture rotary endodontic instruments contain three microstructural phases (austenite, martensite and R–phase) and the traditional NiTi rotary instruments
are in the superelastic austenitic condition (Brantley et al, 2002; Kuhn et al, 2002).

Despite the wide acceptance of NiTi endodontic instruments, the incidence of clinical fracture is about five to seven percent, and the fractured segments left in the root canal may have serious consequences for patients (Parashos et al, 2006). Therefore considerable research has been focused on improving the fracture resistance of NiTi rotary instruments. Surface modifications such as ion implantation have been reported to improve the cutting efficiency and the surface hardness of these instruments (Li et al, 2007). Thermal nitridation and nitrogen-ion implantation treatment of NiTi files were applied to yield a higher N:Ti ratio and increased cutting ability (Rapisarda et al, 2000). Although these surface treatments have obvious advantages for improving fatigue resistance and cutting efficiency of NiTi endodontic instruments, their high cost limits wide usage among manufacturers.

Recently, NiTi rotary instruments (GT® Series X™, Dentsply Tulsa Dental Specialties) made from a NiTi wire subjected to proprietary thermomechanical processing (M-Wire) have been introduced into the market. The manufacturer claims that these instruments have superior flexibility and fatigue resistance than conventional NiTi rotary instruments made from superelastic wire. Clinical use has proved the superior performance of these M-Wire instruments (Dr. John Nusstein, private communication). However, at this time there are no published articles on the cutting efficiency and fatigue resistance of the M-Wire instruments, and the fundamental mechanism for this improved clinical performance is
unknown. Since the microstructure and phase transformation behavior determines the mechanical properties of NiTi alloys, study of the phase transformations could provide significant information for the new M-Wire instruments.

Previous studies (Brantley et al, 2002b and 2002c) suggested that the structure of conventional NiTi rotary instruments could be conveniently investigated by differential scanning calorimetry (DSC). Structural transformations in the NiTi alloys are revealed as endothermic peaks on the heating curves and as exothermic peaks on the cooling curves. Although X-ray diffraction analysis is a useful method to investigate the structure of NiTi alloy (Thayer et al, 1995), this technique only reveals the structure within approximately 50 \( \mu \)m of the surface, whereas DSC provides information for the overall bulk specimen (Brantley, 2001) and the effects of temperature changes on the phase transformations are easily studied. DSC study of conventional ProFile\textsuperscript{®} and Lightspeed\textsuperscript{™} nickel-titanium rotary endodontic instruments in both the as-received condition (Brantley et al, 2002b) and after simulated clinical use (Brantley et al, 2002c) has shown that both instruments exist in the superelastic austenite condition at room temperature.

Previous study of segments of the starting M-Wire for rotary instrument found that the \( A_f \) temperature (45°C – 50°C) is much higher than that for conventional superelastic wire (approximately 20°C or lower) for rotary instruments (Alapati et al, 2008). The purpose of this study was to employ DSC to study the NiTi phase transformations over a range of temperatures for as-
received and clinically used GT® Series X™ instruments made from M-Wire, and thus to examine the effect of manufacturer fabrication of the M-Wire instruments from the starting wires and the effect of clinical use on the NiTi phase transformations.

2.2 Materials and methods

As-received GT® Series X™ instruments (size 30 with .04 taper) were received from the manufacturer (Dentsply Tulsa Dental Specialties). Clinically used (7-8 times) GT® Series X™ instruments were collected from the Graduate Endodontic Clinic of the College of Dentistry at The Ohio State University. Three size/taper combinations were selected for clinically used M-Wire instruments in this study: size 20 with .04 taper, size 30 with .04 taper, and size 40 with 0.08 taper. Instruments were 25 mm in length. Representative test specimens (3 samples of as-received GT® Series X™ instruments from the same batch, 2 samples of clinically used GT® Series X™ instruments size 20 with .04 taper, and 1 sample each for size 30/.04 taper and size 40/.08 taper) for DSC analyses were carefully cut from each instrument using a water-cooled, slow-speed diamond saw. Each test specimen was a single segment of 4 to 5 mm length. The first segment included the instrument tip (weighing 1 – 2 mg), and the other two segments (each weighing 2 – 9 mg) were cut from adjacent portions (termed middle and shank) of the shaft. Each test specimen was placed in an open aluminum pan, following the method used in previous studies (Brantley et al, 2002b and 2002c). An empty aluminum pan served as the control specimen for
the DSC study. The DSC analyses were conducted (Model Q100, TA Instruments, Wilmington, DE) over a temperature range from -80°C to 150°C. For each analysis, the specimen was first cooled from room temperature to -80°C, then heated to 150°C to obtain the heating DSC curve, and subsequently cooled from 150°C back to -80°C to obtain the cooling DSC curve. The linear heating or cooling rate was a standard 10°C/min (Brantley et al, 2002b and 2002c), and during each analysis the DSC cell was purged with dry nitrogen at a rate of 50 mL/min. The plots were analyzed by computer software (TA Universal Analysis 2000) to obtain the onset temperatures for the phase transformations, along with the enthalpy changes (ΔH) associated with these processes. Interpretations of the plots were based on previous DSC studies of two brands of rotary endodontic instruments in the as-received condition (Brantley et al, 2002b) and after simulated clinical use (Brantley et al, 2002c).

2.3 Results

Table 2.1 shows a comparison of the heating and cooling transformation temperatures and enthalpy changes determined from the DSC plots for GT® Series X™ size 30 instruments with .04 taper in the as-received and clinically used conditions. The differences in transformation temperatures are similar for both conditions, indicating that there is minimal effect from 7 to 8 times of clinical use.

Table 2.2 presents a summary of the results from the DSC analyses of the clinically used (7 - 8 times) GT® Series X™ instruments of three different sizes.
There were two used instruments in each case for size 20 with .04 taper, size 30 with .04 taper, and size 40 with 0.08 taper. For all of these instruments, the first specimen included the tip, and the second and third specimens were cut from the adjacent 4 – 5 mm lengths of the shaft. It can be seen that there was minimal effect of the size-taper combination for the used instruments on the transformation temperatures and enthalpy changes.

Figure 2.1 presents the DSC plots for the tip segment from an as-received size 30 GT® Series X™ instrument with .04 taper. A double endothermic peak on the heating (lower) curve, with an onset temperature of 27°C and enthalpy change (ΔH) of 2.4 J/g, corresponds to the initial transformation from martensite to R-phase (weaker peak on left shoulder), followed by transformation from R-phase to austenite (main higher-temperature peak). The single peak on the cooling (upper) curve, with an onset temperature of 44°C and an enthalpy change of 2.0 J/g, corresponds to the transformation from austenite to martensite and may include an unresolved initial transformation from austenite to R-phase.

Figure 2.2 shows the DSC plots for all three segments from the as-received size 30 instrument with .04 taper in Figure 2.1. The two peaks on the heating curve represent the transformation from martensite to R-phase, followed by transformation from R-phase to austenite phase. The austenite-finish (A_f) temperature is similar to that in Figure 2.1 and is above 40°C. As in Figure 2.1, the single peak on the cooling curve may represent the direct transformation from austenite to martensite, or consist of two unresolved peaks corresponding to the initial transformation from austenite to R-phase followed by subsequent
transformation from R-phase to martensite. The latter interpretation is suggested by the long lower-temperature shoulder on this asymmetric peak.

Figure 2.3 presents the DSC plots for the tip segment from the size 30 GT® Series X™ instrument with .04 taper that was subjected to 7 – 8 times of clinical use. The transformation onset temperatures for peaks in these DSC plots were similar to those shown in Figure 2.1 for the tip region of the as-received instrument of the same size and taper, except that the enthalpy changes in the tip region for the clinically used instrument are much lower than for the as-received condition.

Figure 2.4 shows the DSC plots for the tip segment of the GT® Series X™ instrument size 20 with .04 taper after 7 – 8 times of clinical use. There is a single endothermic peak on the heating (lower) curve, with an onset temperature of 37°C and an enthalpy change (ΔH) of 8.1 J/g, for the transformation from martensite to austenite. The A_f temperature for this tip segment is over 50°C. The single exothermic peak on the cooling (upper) curve, with an onset temperature of 43°C and an enthalpy change of 1.8 J/g, corresponds to the transformation from austenite to martensite. The asymmetric shapes of both peaks suggest that the heating and cooling transformations may involve the intermediate R-phase.

Figure 2.5 shows the DSC plots for the three segments of the GT® Series X™ instrument size 20 with .04 taper in Figure 2.4 that was subjected to 7 – 8 times of clinical use. The values of the transformation temperatures and enthalpy changes are given in Table 2.2. The ΔH values are similar to those for the three segments of the as-received size 20 instruments in Figure 2.2.
Figure 2.6 shows the DSC plots for the tip segment from the size 40 GT® Series X™ instrument with 0.08 taper that was subjected to 7 – 8 times of clinical use. The values of the onset temperatures and enthalpy changes for the transformations are given in Table 2.2. The ΔH values of approximately 1 J/g lie in the range for the tip region of the clinically used size 30 instrument with .04 taper in Figure 2.3.

2.4 Discussion

The DSC results in Tables 2.1 and 2.2 and Figures 2.1 and 2.2 show that the as-received GT® Series X™ rotary endodontic instruments have austenite-onset (A\textsubscript{S}) temperatures for the beginning of the transformation from martensite to austenite above room temperature (25°C), and austenite-finish (A\textsubscript{F}) temperatures above 40°C. While these results suggest that as-received GT® Series X™ rotary endodontic instruments made from M-Wire would be essentially in the martensite condition at room temperature, it is important to emphasize that stable martensite which does not undergo transformation over the temperature range analyzed by DSC will not be detected. The presence of such substantial stable martensite in the M-Wire microstructure is suggested by the generally low ΔH values compared to those found for superelastic orthodontic wires (Brantley et al, 2002a and 2003). The presence of a martensitic microstructure for M-Wire at room temperature is confirmed by our complimentary metallographic examination, which will be discussed in Chapter 4. While a previous DSC study (Brantley et al, 2002b) of as-received ProFile®
suggested that conventional NiTi rotary instruments have a completely austenitic structure at room temperature, metallographic examination of the wire blank segments, also discussed in Chapter 4, indicate that these instruments also have a substantially martensitic structure at room temperature.

The results for the tip segment and three segments of GT® Series X™ instruments presented in Figures 2.1 – 2.6 show that there is no evident effect of clinical use on the transformation from martensite to austenite. The same results were found in a previous study on used ProFile® (Dentsply Tulsa Dental) and Lightspeed™ (Lightspeed Technology) conventional NiTi rotary instruments (Brantley et al, 2002c).

Single-segment specimens were used in the present research, as in previous studies (Brantley et al, 2002b and 2002c), to evaluate the possible effects of variations in processing by the manufacturer along the instrument axis on the phase transformations in the M-Wire instruments. Figures 2.1 to 2.6 show that satisfactory DSC plots were obtained with the relatively low-mass segments, although sloping baselines with some noise generally occurred, particularly with the tip segment from the size 30 GT® Series X™ instrument with .04 taper. One broad endothermic peak was observed in most specimens during the heating cycle, corresponding to transformation from martensite to austenite, and one broad exothermic peak was observed during the cooling cycle, corresponding to direct transformation from austenite to martensite. However, it has been noted
that the asymmetric nature of these peaks suggests that the intermediate R-phase may form during both the heating and cooling transformations.

Table 2.1 suggests that there were moderate differences in transformation temperatures and enthalpy changes for the three test segments from each instrument arising from variations in stress and permanent deformation along the instrument axis during the manufacturing process or to differences in local stress along the axis during the clinical instrumentation. The tip region was expected to experience the greatest mechanical stress during manufacturing of the instrument and subsequent clinical use in the form of work hardening of the NiTi alloy. This hypothesis was confirmed in the present study by the higher austenite-start ($A_s$) and austenite-finish ($A_f$) temperatures of the tip segment for nearly every instrument that was analyzed by DSC.

2.5 Conclusions

In conclusion, our DSC study suggested that at room temperature GT® Series X™ instruments made from M-Wire exist in the martensite phase, in contrast to conventional NiTi rotary instruments fabricated from superelastic austenite wire. This martensite phase results from extensive thermomechanical processing that occurs during the manufacture of the starting M-Wire segments. The special martensite structure accounts for the superior mechanical properties of M-Wire instruments, as will be discussed in chapters to follow. It is important to emphasize that DSC only detects NiTi phases that undergo transformation with
temperature changes and that the extensive stable martensite in M-Wire instruments will not generate strong peaks on the heating and cooling curves.
Table 2.1 Comparison of properties determined from DSC plots for two GT® Series X™ instruments of size 30 with .04 taper for both the as-received and clinically used conditions. (Mean values of measured properties are shown.)

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<thead>
<tr>
<th></th>
<th>As-Received</th>
<th>Clinically Used</th>
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<tr>
<td></td>
<td>Tip</td>
<td>Middle</td>
</tr>
<tr>
<td>Heating</td>
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<tr>
<td>$A_s$ (°C)</td>
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<td>28.4</td>
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<td>$A_r$ (°C)</td>
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<td>Cooling</td>
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<td>$R_s$ or $M_s$ (°C)</td>
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<td>43.4</td>
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<td>$\Delta H$ (J/g)</td>
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<td>2.8</td>
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</table>

*When one peak is observed on heating, $A_s$ and $\Delta H$ correspond to the onset temperature and the overall enthalpy change for transformation from martensitic NiTi to austenitic NiTi.

†Onset temperature for transformation from austenitic NiTi to R-phase or for transformation from austenitic NiTi to martensitic NiTi, on cooling. The enthalpy change corresponds to the overall transformation to martensitic NiTi.
Table 2.2. Properties determined from DSC plots for GT® Series X™ instruments after seven to eight times of clinical use (2 samples of clinically used M-Wire instruments size 20 with .04 taper, and 1 sample each for size 30/.04 taper and size 40/.08 taper). The same convention for transformation temperatures and enthalpy changes in Table 2.1 is used in this table as well.
Figure 2.1  DSC heating (*lower*) and cooling (*upper*) curves for the tip region of an as-received GT® Series X™ instrument size 30 with .04 taper.
Figure 2.2  DSC heating (lower) and cooling (upper) curves for the three segments of the as-received GT\textsuperscript{®} Series X\textsuperscript{™} instrument size 30 with .04 taper in Figure 2.1.
Figure 2.3 DSC heating (*lower*) and cooling (*upper*) curves for the tip region of the clinically used GT® Series X™ instrument of size 30 with .04 taper.
Figure 2.4  DSC heating (lower) and cooling (upper) curves for the tip segment of the clinically used GT® Series X™ instrument of size 20 with .04 taper.
Figure 2.5  DSC heating (lower) and cooling (upper) curves for the three segments of the clinically used GT® Series X™ instrument size 20 with .04 taper.
Figure 2.6  DSC heating (lower) and cooling (upper) curves for the tip region of the clinically used GT® Series X™ instrument size 40 with .08 taper.
CHAPTER 3

VICKERS HARDNESS STUDY OF M-WIRE INSTRUMENTS

Abstract

Hardness can be used to predict wear resistance and the ability of NiTi rotary endodontic instruments to abrade tooth structure. Rotary instruments fabricated from M-Wire have superior fatigue resistance compared to instruments fabricated from conventional superelastic wire, but no hardness measurements on M-Wire instruments have been reported. This study employed the Vickers hardness test to gain insight into mechanical properties of M-Wire instruments. As-received and clinically used M-Wire instruments were cut with a diamond saw into 3 segments: tip region, intermediate segment, and third segment, with each segment about 4 – 5 mm in length. Segments were resin-mounted and metallographically polished, and Vickers hardness measurements were performed at room temperature using a 300 g load. Five indentations were made at the center and equidistant adjacent locations in each segment, and diagonal lengths were measured with an optical microscope. Mean hardness values of M-Wire instruments were compared with values for conventional ProFile®
instruments using ANOVA, and the Tukey multiple range test with \( P < 0.05 \) for statistical significance. The mean Vickers hardness for the tip, intermediate and third segments of size 30 / .04 taper M-Wire instruments were significantly \( (P < 0.01) \) higher than those for the conventional NiTi rotary instruments. The hardness at the tip region was significantly \( (P < 0.05) \) lower than at the third segment for each M-Wire instrument. No significant effect on hardness was found following clinical use. Combined with our DSC and microstructure studies of this new M-Wire rotary instrument, we concluded that the increased hardness of this material results from its martensitic microstructure and strengthening mechanisms induced by the extensive thermomechanical processing. The improved clinical performance of M-Wire instruments may be attributed to this increased hardness.

### 3.1 Introduction

Rotary instruments machined from NiTi have been widely used in dental practice due to the special properties of this alloy: much lower modulus of elasticity compared with stainless steel and superelastic behavior (Brantley et al, 2001). Although NiTi rotary instruments have the ability to prepare curved root canals, occasional instrument fracture does occur (Paraschos et al, 2006).

Considerable research has been focused on improving the cutting efficiency and cyclic fatigue resistance of the rotary instruments using ion implantation (Lee et al, 1996; Rapisarda et al, 2001; Li et al, 2007) and thermal treatment (Zinelis et al, 2007). Ion implantation has been proven to be an
effective method for increasing surface hardness and wear resistance of NiTi instruments. Other surface modification methods, such as physical vapor deposition and dry cryogenic treatment, have also been applied to yield improved cutting efficiency and fatigue resistance, which the authors attributed to increased hardness on the instrument surface (Rapisarda et al, 2000; Schäfer, 2002; Vinothkumar et al, 2007). Therefore, the surface hardness of a rotary instrument is considered to be closely related to its fatigue resistance and cutting efficiency.

Recently, a new NiTi Wire named “M-Wire” and processed by a novel thermomechanical procedure (Sportswire LLC, Langley, OK) has been developed for endodontic rotary instruments. Studies have shown that this new M-Wire has improved fatigue resistance compared to the superelastic wire used to manufacture conventional NiTi rotary instruments (Private communication, William Ben Johnson, Sportswire LLC). Studies of M-Wire by our group have found that the thermomechanical processing for M-Wire yields much higher $A_t$ temperatures than for conventional superelastic orthodontic wires and that the superior mechanical properties of M-Wire arise from the special character of the martensite in its microstructure, which is different from the microstructure of the conventional NiTi superelastic wires for rotary instruments (Alapati et al, 2008; Brantley et al, 2008, Liu et al, 2009). NiTi rotary instruments fabricated from this M-Wire are marketed by Densply Tulsa Dental Specialties. The manufacturer has claimed that the M-Wire rotary instruments (GT® Series X™) have superior fatigue resistance and cutting efficiency compared to instruments fabricated from
conventional superelastic wire, but no articles reporting the mechanical properties of M-Wire instruments have been published to date.

A study of clinically used conventional NiTi rotary instruments (Alapati et al, 2006) has shown that the mean Vickers hardness number (HV) for the tip region (D2 – D4) ranged from 313 to 324, and these values were similar to the previously reported Vickers hardness of a shape memory NiTi orthodontic wire (Brantley, 2001). Alapati et al (2006) also found that the mean Vickers hardness of the used instruments was greater in the D6 – D10 region compared to the tip region and still higher at the D14 position and beyond (closer to the instrument shank). However, no data on the hardness of M-Wire and M-Wire instruments have been reported. As hardness is related to two highly important mechanical properties of rotary instruments, cutting efficiency and fatigue resistance, hardness measurements could potentially provide valuable information on the performance of the new M-Wire instrument.

The purpose of this study was to investigate the hardness of M-Wire instruments for comparison with NiTi rotary instruments fabricated from conventional superelastic wire. The effect of clinical use on the hardness of M-Wire instruments was also examined.

3.2 Materials and Methods

As-received M-Wire instruments (GT® Series X™) were provided by Dentsply Tulsa Dental Specialties. M-Wire and ProFile® instruments that had experienced 7 – 8 clinical uses in Faculty Practice of the College of Dentistry
were also provided. All clinically used instruments had no visible evidence of permanent torsional deformation. In addition to the instruments, segments from one batch of M-Wire used to manufacture M-Wire rotary instruments (provided by Sportswire LLC) and one batch of 35°C Copper NiTi wire (Ormco) used for orthodontic treatment were selected for study. Five samples in each group (as-received M-Wire instruments, and clinically used M-Wire and ProFile® instruments) were cut perpendicularly to the long axis of the instrument with a water-cooled diamond saw into 3 adjacent segments: tip region, intermediate segment, and shank region. Each segment was about 4 – 5 mm in length. The M-Wire and copper NiTi blanks were also cut into segments about 5 mm in length. These segments were embedded in acrylic metallographic resin and subjected to a standard sequence of metallographic preparation. A polishing machine (Metaserv 2000, Buehler UK Ltd, Coventry, West Midlands, England) was used to grind and polish the surfaces of the mounted specimens with sandpaper (Carbimet Paper Discs, Buehler: 240 and 600 grit), followed by alumina paste (Alpha Micropolish, Buehler: 6 µm, 1 µm and 0.5 µm particle sizes).

Vickers hardness measurements were performed at room temperature with 300 g load and 15 s dwell time (Micromet 2100, Buehler). Five indentations were made at the center and at equidistant adjacent locations in each segment. By observing the sample under the optical microscope at lower magnification, it was ensured that the distance between each indentation was larger than at least ten indentation widths, so the effect of previous loading on the hardness of the
adjacent loading area would be minimized. Diagonal lengths were measured with the optical microscope associated with the microhardness tester. The Vickers hardness number (VHN) was obtained from the equation relating HV, indenting load, and mean diagonal length (Dieter, 1986).

Three groups of comparisons were made: (1) the means of the hardness measurements in the three regions of the new GT® Series X™ instruments were compared; (2) the means of the hardness measurements in the three regions of the new and used GT® Series X™ instruments were compared; and (3) the means of the hardness measurements in the three regions of the used GT® Series X™ instruments were compared to values obtained for used ProFile® conventional NiTi rotary endodontic instruments. For these comparison, one-way ANOVA was used, followed by the Tukey multiple range test, with P < 0.05 for statistical significance.

3.3 Results

Figure 3.1 shows a representative Vickers hardness indentation on a flute of a resin-mounted NiTi rotary instrument. Figure 3.2 shows that the mean Vickers hardness numbers for the tip, intermediate and shank regions of size 30 / .04 taper GT® Series X™ M-Wire instruments were 374, 380 and 392, respectively. These values are considerably higher than previously reported Vickers hardness for clinically used conventional ProFile® GT® NiTi instruments (Alapati et al, 2006). The Vickers hardness at the third segment is significantly
higher than at the tip region and the intermediate segment for as-received instruments (P < 0.05).

Figure 3.3 shows the Vickers hardness for the as-received and clinically used GT® Series X™ M-Wire instruments. No significant difference (P<0.05) was found when the same segments were compared for the as-received and used instruments.

Figure 3.4 compares the Vickers hardness numbers for the tip, intermediate and third segments of clinically used GT® Series X™ M-Wire instruments and conventional ProFile® NiTi rotary instruments. The hardness for each segment in the clinically used M-Wire instruments was significantly higher (P<0.01) than the corresponding segment in the clinically used conventional instruments.

Figure 3.5 compares the Vickers hardness numbers for the M-Wire blanks and as-received M-Wire instruments. The hardness of the M-Wire instruments was significantly higher than the M-Wire blanks used for manufacture of the M-Wire instruments.

3.4 Discussion

Previous studies have shown that the surface hardness of NiTi rotary instruments contributes significantly to their cutting ability and wear resistance during canal instrumentation (Lee et al, 1996; Rapisarda et al, 2000; Schäfer et al, 2002; Li et al, 2007; Vinothkumar et al, 2007). Canal instrumentation is a complex procedure characterized by self-threading and abrasive effects. The top
Dentin is formed into chips by shearing force from the instrument, and then reduced in size to small chips by a combination of cutting and friction mechanisms (Shen et al, 2008). It is thus plausible that increased NiTi instrument hardness due to surface modification has previously been found to yield improved fatigue resistance and cutting efficiency (Schäfer, 2002; Vinothkumar et al, 2007). In the present study, because significantly increased hardness was found for M-Wire instruments compared with conventional ProFile® instruments, it is expected that M-Wire instruments would have better cutting ability and wear resistance.

Various methods have been suggested to change the mechanical properties of the NiTi alloy used for rotary instruments (Thompson, 2000). Thermomechanical processing is the most widely used method by manufacturers, and a study has shown that the mechanical properties of NiTi rotary instruments are strongly dependent on the thermomechanical processing history of the starting NiTi wire alloys (Johnson, 2009). Typical processing of superelastic NiTi wire for rotary endodontic instruments includes vacuum casting and hot forging, rolling, and drawing followed by heat treatment between 450°C and 550°C (Thompson et al, 2000; Young et al, 2005; Johnson, 2009). Heat treatment or thermal processing can significantly modify the mechanical properties of NiTi alloys and rotary instruments (Hamanaka et al, 1989; Liu et al, 1994; Kuhn et al, 2002; Frick et al, 2005). For example, heat sterilization of rotary instruments at 170°C for 1 h up to 5 times significantly increased the microhardness and fatigue resistance of ProFile® and Quantec instruments, but the absence of an effect of
one-time sterilization on mechanical properties suggests that the effect of heat
treatment is related to the heating time period (Serene et al, 1995; Chaves
Craveiro de Melo et al, 2002). In other studies, increased fatigue resistance of
NiTi files was found after heat treatment between 440°C and 550°C, but
decreased surface hardness was observed (Kuhn et al, 2002; Zinelis et al, 2007).
These authors suggested that the loss of work hardening effects because of the
heat treatment accounted for the observations.

Recently a new NiTi rotary instrument was introduced in which the starting
NiTi wire blanks are twisted rather than machined (Twisted File [TF],
SybronEndo). According to the manufacturer, the microstructure of TF
instruments consists of R-phase (accomplished by appropriate
thermomechanical processing). Recent research by Gambarini et al (2008) has
shown that the TF instrument is significantly more flexible than the ProFile®
(Dentsply Maillefer) instrument of the same taper and tip size.

Although the thermomechanical treatment history for the M-Wire
instruments is not clear, our complimentary studies described in other chapters
have provided information about their microstructures and phase transformations.
Use of DSC on segments of starting M-Wire segments and instruments, STEM
examination of foils prepared from M-Wire segments, and metallographic
examination of etched microstructures for both M-Wire segments and
instruments have revealed a special stable martensite structure at room
temperature that accounts for the improved mechanical properties of M-Wire and
the GT® Series X™ rotary instruments. STEM observations of extensive slip
bands and microtwins, which serve as strengthening mechanisms for this martensite structure, account for the higher Vickers hardness of the M-Wire instruments found in this study.

The hardness of clinically used M-Wire instruments appeared to be slightly increased compared with the same segments of as-received M-Wire instruments, if Figure 3.3 is examined closely. This may be due to the work hardening during clinical use procedure. However, no statistically significant difference (P<0.05) was found. The absence of an evident effect of clinical use on Vickers hardness of M-Wire rotary instruments is consistent with a previous study of the Vickers hardness for clinically used conventional rotary NiTi instruments (Alapati et al, 2006). As in that study, the tip region of the M-Wire instruments for both the as-received and clinically used conditions was found to have the lowest Vickers hardness compared with the adjacent 5 mm region and a third 5 mm region that was further from the tip. The Vickers hardness (353 HV) of the M-Wire blanks, which are used for the manufacture of M-Wire instruments, is significantly higher than the hardness of the superelastic 35°C Copper NiTi orthodontic wire (300 HV). However, it is significantly lower than the hardness of the as-received M-Wire instruments in our study. This indicates that the M-Wire blank is work hardened during the manufacturing procedure and that the work hardening (Ashby, 1988) created later in the instrument during the manufacturing process dominates further work hardening that occurs in the instrument during clinical use.

While the present Vickers hardness measurement technique has provided highly useful information about the M-Wire blank segments and rotary
instruments, one concern in our study may be better resolved by further evaluations of the hardness by a nanoindenter. Due to the small diameter at the tip region of a NiTi rotary instrument, which varies from 200 µm to 400 µm, the hardness at the tip may be volume-sensitive. The diagonal length of the tip of the nanoindenter is several micrometers, which is much smaller compared with the microhardness tester. Moreover, the loading force of nanoindenter could be less than 1 gram, which will greatly reduce the effect of material volume on its hardness. Therefore, the use of a nanoindenter will permit more precise examination of the hardness variation along the surface and over the cross-section of the NiTi rotary instruments. This research is currently in progress.

Another important area for future research is an examination of the cutting ability of the M-Wire instruments to ascertain whether there is a correlation of this highly important property with the significantly increased Vickers hardness compared to that of conventional rotary instruments. However, such studies can yield controversial results since there is no standardized methodology for evaluation of cutting ability. For example, bovine bone has been used to simulate human tooth dentin for instrumentation (Machian et al, 1983; Yguel-Henry et al, 1990), and its advantages as a model material for such studies have been discussed (Pruett et al, 1997). However, other investigators preferred to use clear resin blocks with simulated root canals having the curvatures of the actual root canals, which allows more realistic comparison of the shaping ability of rotary instruments (Schäfer et al, 1995 and 2006). However, these resin blocks may be softened by heat generated during the in vitro instrumentation, leading to
instrument binding and separation in laboratory studies (Thompson et al, 2000). Furthermore, the definition of cutting ability varies (Felt et al, 1982; Tepel et al, 1995; Schäfer et al, 2008). This is a considerable problem for the design of a clinically relevant study. At present the most accepted parameter for evaluation of cutting ability is the weight of dentin or the model material removed during instrumentation, but cutting depth has also been measured (Lugassy et al, 1968). In a recent study, Shen et al (2008) recommended the use of microcomputed tomography to provide an evaluation of the cutting ability of NiTi rotary instruments.
3.5 Conclusions

M-Wire instruments have significantly higher hardness compared with conventional rotary NiTi instruments. For both as-received and clinically used instruments, the shank region located more than 10 mm from the tip has the highest Vickers hardness compared to the adjacent 5 mm region along the instrument axis and the 5 mm region that includes the instrument tip. No significant effect of clinical use on the Vickers hardness was found for segments from these three regions of M-Wire instruments. The increased hardness of the M-Wire instruments is attributed to the special work-hardened martensite structure found by STEM examination of starting wire blanks and metallographic examination of the etched microstructures of instruments. This increased hardness is related to the improved mechanical properties of the M-Wire instruments and may provide improved cutting efficiency, although this remains to be established in future studies.
Figure 3.1  Vickers hardness indentation on flute of resin-mounted and polished M-Wire NiTi rotary instrument.
Figure 3.2 Comparison of Vickers hardness for three segments of as-received (new) M-Wire instruments (* denotes significant difference with $P < 0.05$).
Figure 3.3  Comparison of Vickers hardness for as-received and clinically used M-Wire instruments. No significant difference was found for each segment of the new and used instruments.
Figure 3.4  Comparison of Vickers hardness for clinically used M-Wire instruments and conventional ProFile\textsuperscript{®} instruments (* denotes significant difference with P < 0.01).
Figure 3.5  Comparison of Vickers hardness for M-Wire blanks and M-Wire instruments (* denotes significant difference with P < 0.01).
CHAPTER 4

MICROSTRUCTURE STUDY AND WEAR RESISTANCE ANALYSIS OF M-WIRE INSTRUMENTS

Abstract

Properties of NiTi rotary endodontic instruments depend on their microstructural phases. The starting M-Wire for rotary instruments has superior fatigue resistance compared to the superelastic (SE) NiTi wire for conventional rotary instruments. Although the manufacturer claims that M-Wire instruments have superior clinical performance than conventional instruments, no articles reporting the microstructures and mechanical properties of M-Wire instruments have been published. In this study, an etching procedure was employed to reveal the microstructures of as-received and clinically used M-Wire instruments. The instruments were cut into segments, resin-mounted, polished and acid-etched to yield high-quality microstructures. Photomicrographs were obtained with an optical microscope and scanning electron microscope (SEM). Also, bright-field images of foil specimens prepared from M-Wire blanks and conventional
segments were obtained with the scanning transmission electron microscope (STEM). Lastly, the wear resistance of clinically used M-Wire instruments was investigated from comparison to the surfaces of as-received (unused) instruments using an SEM.

Optical microscope images showed that M-Wire instruments had room-temperature martensite microstructures consisting of colonies of lenticular features. STEM images revealed that the martensite structure of the starting M-wire has relatively coarse grains with internal microtwins and deformation bands, as well as triple-point junctions of grain boundaries. These features provided evidence of substantial mechanical deformation and annealing during the wire processing that were not found in STEM images of the conventional SE wire. SEM images indicated that there were less post-instrumentation defects on M-Wire instruments compared with conventional instruments, suggesting higher wear resistance for the M-Wire instruments.

In conclusion, M-Wire instruments appear to have superior clinical wear resistance compared to conventional rotary instruments. The superior mechanical properties of these instruments are attributed to the special martensite structure induced by extensive thermomechanical processing that is not present in conventional NiTi rotary instruments.

4.1 Introduction

The nickel–titanium alloy used for rotary endodontic instruments was first developed in the 1960s. Based on the equiatomic intermetallic compound NiTi,
this alloy composition is about 55% nickel and 45% titanium (wt. %) (Thompson, 2000). There are three major phases of the NiTi alloy: austenite, martensite and R-phase. Transformation between austenite and martensite occurs by twinning, and this process is reversible; R-phase may form as an intermediate structure (Brantley, 2001). The phase transformation and microstructure characteristics determine the mechanical properties of the NiTi alloys (Brantley, 2001) and rotary instruments.

NiTi rotary instruments are machined from starting wire blanks that are in the superelastic condition, which is induced by vacuum casting and hot forging, rolling, and drawing, followed by heat treatment between 450°C and 550°C (Thompson, 2000; Young et al., 2005; Johnson, 2009). DSC study has confirmed that as-received NiTi rotary instruments are in the superelastic (SE) austenite condition (Brantley, 2001), although stable non-transforming martensite would not be detected.

Due to the lower elastic modulus and thus higher elastic flexibility compared with instruments made from stainless steel, NiTi rotary endodontic instruments are widely used in dental clinic because of their ability to negotiate curved root canals. However, intracanal instrument fracture (separation) is still a challenge during endodontic treatment. Serious consequences due to intracanal fracture may include failure of the endodontic treatment, retroperiapical surgery, and even tooth extraction (Parashos et al., 2006). Therefore several studies have focused on strategies to improve the fatigue resistance of rotary instruments (Lee et al., 2000; Rapisarda et al., 2000; Schäfer et al., 2002; Vinothkumar et al., 2007).
Wear resistance of rotary instruments can be evaluated at high magnification from SEM observations of instrument surfaces (Eggert et al, 1999; Vinothkumar et al, 2007). Manufacturer defects in as-received instruments or defects induced during canal preparation may serve as crack initiation sites, and crack propagation induced by stress concentration at the defects will significantly decrease the fatigue resistance of rotary instruments, leading to separation (Askeland et al, 2003). Therefore, clinical wear resistance is highly desired for rotary instruments.

Recently a new rotary NiTi instrument (GT® Series X™), machined from M-Wire blanks, processed by a proprietary thermomechanical processing technique (Sportswire LLC, Langley, OK), has been marketed (Dentsply Tulsa Dental Specialties). Laboratory studies (Sportswire) have shown that the M-Wire has a higher ratio of tensile strength to upper superelastic plateau stress, compared with the superelastic wire used to manufacture conventional rotary instruments. Although the new M-Wire rotary instruments are claimed to have better clinical performance than conventional NiTi rotary instruments, there have been no published reports on the clinical performance of these instruments.

The principal aim of this study was to characterize the etched microstructure of M-Wire instruments by using an optical microscope and an SEM with energy-dispersive spectrometric analyses. The wear resistance of M-Wire instruments was investigated by comparing the surface defects on clinically used and as-received instruments with conventional NiTi rotary instruments. Scanning transmission electron microscope (STEM) images of the starting M-Wire
segments and conventional superelastic wire segments were obtained in a complementary study to provide detailed information about the ultrastructure that would not be possible with the optical microscope or SEM.

4.2 Materials and methods

4.2.1 Materials

As-received M-Wire instruments (GT® Series X™) size 30/.04 taper were provided by Dentsply Tulsa Dental Specialties. Clinically used (7 – 8 times) GT® Series X™ instruments were collected from Faculty Practice in the College of Dentistry, The Ohio State University (provided by Dr. John Nusstein). All clinically used M-Wire instruments had no visible evidence of permanent torsional deformation. Clinically used ProFile® rotary instruments (Dentsply Tulsa Dental Specialties) with and without fractured tip regions were also provided by Dr. Nusstein.

4.2.2 Wear resistance

As-received and clinically used M-Wire instruments (GT® Series X™) Instruments selected for SEM observation were first agitated ultrasonically for 2 minutes in distilled water, then rinsed with ethanol, and blow-dried in an air stream. Scanning electron microscope (SEM) observations (Quanta, Philips) were made in the secondary electron mode for each instrument. Photomicrographs of the surface defects due to manufacture processing for new instruments and of surface wear in used instruments due to clinical use were obtained for qualitative visual evaluation (Vinothkumar et al, 2007).
4.2.3 Microstructures

As-received and clinically used M-Wire instruments (GT® Series X™) selected for microstructural observations were cut perpendicularly to the long axis of the instrument with a water-cooled diamond saw into 3 segments (tip region, intermediate segment, and shank region), with each segment about 4 – 5 mm in length. In addition to the instruments, segments from one batch of M-Wire and one batch of conventional SE NiTi wire (Maillefer) used to manufacture rotary instruments (provided by Sportswire LLC) were selected for study. The wires were cut into 5 mm segments. Specimens were embedded in acrylic resin to reveal the horizontal surface along the axis and the perpendicular cross-section. All specimens were polished with a standard sequence of metallographic abrasives (Buehler): sandpaper (240 and 600 grit) and alumina paste (6 µm, 1 µm and 0.5 µm particle sizes). The polished surfaces were etched in a solution (3 mL hydrofluoric acid, 5 mL nitric acid and 20 mL acetic acid) that yielded detailed microstructures. The etched microstructures of the starting NiTi wire and rotary instrument specimens were examined with an optical microscope and an SEM. Magnifications ranging from ×50 to ×5000 were used with the SEM. Compositions of precipitates in the microstructures were obtained by energy-dispersive spectrometric analyses (EDS) with the SEM.

Scanning transmission electron microscope (STEM) (Williams, 1996) observations were performed to investigate the microstructures of the starting NiTi wires at the submicron level. A focused ion beam (FIB) technique was employed to prepare foils from cross-sectioned segments of M-Wire and the
conventional superelastic wire. The Tecnai TF-20 electron microscope (Philips, Eindhoven, The Netherlands) was operated at 200 kV, and bright field images were obtained at a variety of magnifications.

4.3 Results

4.3.1 SEM examination of instruments

Figure 4.1 shows representative images of manufacturing defects observed in the as-received M-Wire rotary instruments. Debris, metal rollover at the edges of the radial lands, and parallel grooves from the machining process were evident on all as-received M-Wire and conventional rotary instruments.

Figures 4.2, 4.3 and 4.4 illustrate the effects of clinical use on the ProFile® rotary instruments. Surface deformation at the machining grooves, elongated pits in the radial lands, and embedded dentin chips are evident.

Figure 4.5 and 4.6 are representative images of clinically used M-Wire instruments. Compared with the clinically used ProFile® instruments, the clinically used M-Wire instruments have shallower areas of surface deformation and fewer embedded dentin chips.

Figures 4.7 and 4.8 show that the fracture surfaces of clinically retrieved ProFile® instruments contain small voids, oxide particles and the characteristic dimpled structure associated with ductile fracture. No fractured M-Wire instruments from the Faculty Practice clinic were available; Dr. Nusstein has not experienced separation of these instruments during root canal therapy for patients.
4.3.2 Light microscope study of etched wires and instruments

Figure 4.9 – 4.12 show the microstructures of M-Wire, conventional SE wire, and M-Wire instruments observed with the optical microscope.

Figure 4.9 illustrates the microstructure of M-Wire for the surface along the wire axis (Figure 4.9a) and for a cross-section perpendicular to the wire axis (Figure 4.9b). Both micrographs exhibit the classical lenticular martensite structure (Reed-Hill and Abbaschian, 1994). Figure 4.10 similarly shows the microstructures of the Maillefer conventional SE wire for the surface along the wire axis (Figure 4.10a) and the cross-section perpendicular to the wire axis (Figure 4.10b). A similar lenticular martensite structure to that found for M-Wire is evident.

Figures 4.11 and 4.12 show the microstructure of M-Wire instruments in the as-received and clinically used conditions. Both conditions for these instruments exhibit the same lenticular martensite structure found for the starting M-Wire segments shown in Figure 4.9. The microstructures at the tip, middle and shank regions of the M-Wire instruments had no obvious differences.

For comparison to the preceding optical microscope photographs, Figure 4.13 presents a secondary electron SEM image of the etched surface of an as-received M-wire instrument. The lenticular martensite structure with substantial twinning is again apparent, and the more distinct appearance of the individual martensite laths is consistent with the much greater depth of focus for the SEM.
4.3.3 STEM study of conventional SE wire and M-Wire

Figure 4.14 presents STEM images of the cross-section of the conventional Maillefer SE wire. The microstructure consists of very fine equiaxed grains of predominantly austenite, with some darker grains which may be R-phase or martensite.

Figure 4.15 presents STEM images of the cross-section of M-Wire. The effects of the extensive thermomechanical processing are apparent. Much coarser grains and triple-point junctions of grain boundaries due to annealing can be seen in Figure 4.15a, and considerable twinning from the extensive mechanical deformation can be seen in Figure 4.15b.

4.3.4 EDS study of precipitates

Figure 4.16 shows the SEM image of a typical, nearly rectilinear, precipitate that is found in the microstructures of M-Wire segments and M-Wire instruments. EDS analysis indicates that the precipitates are titanium-rich, with an approximate composition of Ti$_2$Ni (Brantley et al, 2001; Otsuka et al, 2006).

4.4 Discussion

The HF–HNO$_3$–acetic acid etchant used with the NiTi wires and rotary instruments yielded high-quality microstructures. Initial efforts with the well-known Kroll’s etchant (HF–HNO$_3$–water) did not yield acceptable results, and it was found that addition of the acetic acid component provided excellent control of the etching process. Hydrofluoric acid removes the surface oxide from the NiTi specimen, and nitric acid preferentially attacks the grain boundaries.
Our study has shown that etched blank M-Wire and M-Wire instruments have classic lenticular martensitic microstructures when viewed with the optical microscope at room temperature. These observations are in agreement with results from our DSC study, presented in Chapter 2, which suggested that M-Wire instruments are martensite or a mixture of martensite and R-phase at room temperature. The unexpected observation was that at room temperature the conventional Maillefer SE wire (and presumably the machined rotary instruments) have a similar, largely martensitic microstructure, although DSC analyses indicate that the NiTi alloy has an austenitic microstructure. This apparent paradox is resolved when one realizes that DSC only detects the martensite that transforms to austenite and not stable martensite that does not undergo transformation. Careful examination of Figure 4.10 reveals that some flat, non-lenticular areas exist in the microstructure and correspond to the austenite phase.

The scanning transmission electron microscope (STEM) provided details at the nanometer scale for thin foils of M-Wire that cannot be observed with the SEM or optical microscope. The STEM observations revealed that the extensive thermomechanical processing of M-Wire provides efficient strengthening mechanisms that are not found in conventional superelastic wires for endodontic rotary instruments. The special martensite structure of M-Wire readily accounts for its improved mechanical properties and the excellent clinical performance of the M-Wire instruments.

The wear resistance of M-Wire instruments was qualitatively evaluated by examining the surfaces after clinical use. As would be predicted from the
substantial higher values of Vickers hardness, better wear resistance was found for M-Wire instruments, with less surface defects and deformations of the radial lands compared with clinically used conventional ProFile® instruments. The effect of surface defects on the fatigue life of NiTi rotary instruments has been extensively discussed (Borgula, 2005; Alapati et al, 2005; Cheung et al, 2005 and 2007a). The conventional NiTi rotary instruments are reported to separate as a result of cyclic fatigue, which is likely to occur when the instruments rotate in curved root canals. Crack initiation occurs on the conventional instrument surface, followed by transgranular crack growth (Cheung et al, 2005 and 2007a). Therefore wear resistance is critical to the working life of rotary instruments, and the improved wear resistance of M-Wire instruments suggested by our SEM observations is attributed to the increased hardness of these instruments. In future research, additional information about wear resistance can be obtained by examining the same areas (tip and middle regions) for each instrument with the SEM before and after instrumentation (Vinothkumar et al, 2007).

While fractographic examination of clinically used conventional rotary instruments aids in understanding the origin and direction of crack propagation during fatigue and aims to identify features on fracture surfaces, it has been found that instrument separation is minimal for skilled clinicians. From the present research, the reported excellent tensile mechanical properties and fatigue behavior for M-Wire and the M-Wire rotary instruments is the result of the special martensite structure. It would be interesting to collect some fractured instruments from less experienced clinicians who might inadvertently subject the
instruments to overload and some fractured instruments from more experienced clinicians who used the instruments an excessive number of times. While the nucleation of secondary phase particles in the microstructure of engineering NiTi alloys, such as nickel-titanium oxides was suggested as the main reason for their dimpled fracture surface (Duerig et al, 1990), this may not be a factor for the M-Wire instruments if the metallurgical quality of the NiTi alloy is high. Cyclic fatigue testing of the M-Wire instruments is recommended for future studies.

4.5 Conclusions

A classical lenticular martensitic microstructure was observed for etched M-Wire instruments, and there was no evident difference between the microstructures of the M-Wire instruments for the surface areas along the instrument axis and the cross-sections perpendicular to the axis. Much coarser grains, triple-point junctions of grain boundaries, and heavily microwinned martensite were observed in the microstructures of M-Wire in our STEM study. These features were absent in the microstructures of conventional superelastic wire for rotary instruments, and the strengthening mechanisms for the special martensite structure account for the improved mechanical properties of M-Wire. EDS analyses indicate that the approximate composition of the precipitates in the M-Wire microstructure is Ti$_2$Ni, which indicates that the starting wire segments are Ti-rich rather than Ni-rich. The apparently better wear resistance for M-Wire instruments, suggested by qualitative SEM observations of their surfaces after
clinical use, is consistent with much higher values of Vickers hardness for these instruments, compared to the conventional NiTi rotary instruments.
Figure 4.1 SEM images of as-received M-Wire instruments showing the presence of grooves, metal rollover at the edges of the radial lands, and other surface defects caused by machining the wire blanks.
Figure 4.2 SEM micrograph of a clinically used ProFile® instrument, showing the effects of instrumentation in the root canal on the machining grooves and the radial lands.
Figure 4.3  SEM micrograph of clinically used ProFile® instrument, showing retention of dentin chips in widened machining grooves.
Figure 4.4   SEM micrograph of a clinically used ProFile® instrument, showing a pit on a radial land (near center of image).
Figure 4.5 SEM micrographs of the surface of clinically used M-Wire instrument, showing scratches and surface deformation at lower magnification (a) and higher magnification (b).
Figure 4.6  SEM micrographs of the surface of another clinically used M-Wire instrument at lower magnification (a) and higher magnification (b), showing very shallow microcracks and fewer dentin chips on the instrument surface.
Figure 4.7   SEM micrograph of the fracture surface of a clinically used ProFile® instrument, showing the presence of microvoids (dimpled rupture), along with larger voids and surface debris.
Figure 4.8  SEM micrograph of the fracture surface of a clinically used ProFile® instrument, showing dimpled rupture and the presence of very small oxide particles.
Figure 4.9 Optical microscope photographs of etched M-Wire, showing the microstructure (a) at the surface along the long axis and (b) at a cross-section surface perpendicular the long axis.
Figure 4.10 Optical microscope photographs of the microstructures of etched conventional Maillefer superelastic wire (a) at the horizontal surface along the long axis and (b) at the cross-section surface perpendicular the long axis.
Figure 4.11  Optical microscope photographs of the microstructure of the etched middle region of an as-received M-Wire instrument: (a) surface area along the axis; (b) cross-section surface perpendicular to the long axis; (c) cross-section surface perpendicular to the long axis under much higher magnification.
Figure 4.12 Optical microscope photographs of the microstructures of the etched tip region of a clinically used M-Wire instrument: (a) horizontal surface along the long axis; (b) cross-section surface perpendicular to the long axis.
Figure 4.13 Secondary electron image of etched microstructure of an as-received M-Wire instrument obtained with SEM.
Figure 4.14  Scanning transmission electron microscope (STEM) images of the cross-section of Maillefer SE wire for conventional rotary instruments: (a) lower magnification and (b) higher magnification.

(Dr. Libor Kovarik from the Department of Materials Science and Engineering at The Ohio State University obtained the STEM images in Figures 4.14 and 4.15.)
Figure 4.15  Scanning transmission electron microscope (STEM) images of the cross-section of M-Wire for rotary instruments: (a) lower magnification and (b) higher magnification with optimum two-beam conditions for resolution.
Figure 4.16 SEM micrograph (backscattered electron image) showing a Ti-rich precipitate in the microstructure of an M-Wire instrument.
CHAPTER 5

CONCLUSIONS

In this study various metallurgical laboratory techniques (DSC, Vickers hardness measurements, optical microscope and SEM examination of etched specimens, and STEM examination of foil specimens) were employed to investigate the microstructure and phase transformations for M-Wire and rotary endodontic instruments machined from M-Wire. Information about clinical wear resistance of M-Wire instruments was obtained by SEM examination of used instruments. Under the conditions of the foregoing investigations, the following conclusions can be drawn:

1. M-Wire instruments have much higher \( A_t \) temperatures than conventional superelastic NiTi rotary instruments. The DSC peaks indicate that the M-Wire instruments are largely a mixture of martensite and R-phase at room temperature, with a small amount of austenite. Only small differences are found in the DSC plots for as-received and clinically used instruments.

2. M-Wire instruments have significantly higher Vickers hardness compared with rotary instruments made from conventional superelastic wire. The increased
hardness may contribute to better wear resistance and cutting efficiency for these instruments. The shank region has the highest hardness compared to tip region and middle region. Clinical use had no effect on the Vickers hardness of M-Wire instruments.

3. The M-Wire instruments displayed better wear resistance, along with less permanent deformation of the surface features after 7 – 8 times of clinical use, compared with the conventional ProFile® rotary instruments.

4. A classical lenticular martensite microstructure was observed with the optical microscope and SEM for acid-etched M-Wire and M-Wire instruments. There was no evident difference between the microstructures of surface areas along the wire or instrument axis and cross-sections perpendicular to the axis.

5. STEM examination of M-Wire and conventional SE wire revealed that M-Wire has relatively coarse grains, numerous triple-point junctions of grain boundaries, and localized deformation bands with microtwins, indicative of extensive thermomechanical processing. This efficient use of alloy strengthening mechanisms accounts for the superior mechanical properties of M-Wire compared to conventional superelastic wire for rotary instruments.

6. DSC does not detect the large quantities of stable martensite present in both M-Wire and the conventional SE wire, which are evident in the etched microstructures. The existence of substantial stable martensite is evident from much lower enthalpy changes for transformation from martensite to austenite, compared to superelastic and shape memory NiTi wires for orthodontics.
7. The effect of the manufacturing procedure on the mechanical properties of M-Wire instruments was observed in this study, when the Vickers hardness was compared for the wire blanks and instruments. The manufacturing procedure significantly increases the hardness of M-Wire instruments. However, no effect of the manufacturing procedure on the microstructure of M-wire instruments was observed with the optical microscope for acid-etched samples. Further study by TEM is indicated.


Cheung GS, Shen Y, Darvell BW. Does electropolishing improve the low-cycle fatigue behavior of a nickel-titanium rotary instrument in hypochlorite? J Endod. 2007b;33:1217–21


Engineers edge. http://www.engineersedge.com/material_science/work_strain_hardening.htm


