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Effect of heat sterilization on surface characteristics and microstructure of Mani NRT rotary nickel–titanium instruments

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Abstract

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Aim To evaluate the effect of repeated dry heat sterilization on surface characteristics and microstructure of Mani nickel–titanium rotary instruments.

Methodology Thirty-three new Mani NRT instruments, size 30, taper 0.04 and 25 mm in length were examined. Twenty-seven instruments were divided into three groups for surface characterization by scanning electron microscopy (SEM). In the first group ($n = 3$), instruments were examined in the 'as-received' condition and after they had been subjected to 11 sterilization cycles. In the second and third subgroups ($n = 12$), 12 instruments were prepared for cross-section and a further 12 for longitudinal sectional analysis and evaluated in subgroups of three, after 0, 1, 6 and 11 sterilization cycles. The remaining six instruments were analysed with differential scanning calorimetry (DSC), three in the 'as-received' condition and three after being subjected to 11 cycles of sterilization.

Results Scanning electron microscopy observations indicated the presence of debris, pitting and deep milling marks in both new and sterilized files. After 11 sterilization cycles, debris remained and surface roughness was increased significantly ($P = 0.05$). DSC analyses showed that the specimens in the 'as-received' condition and after 11 sterilization cycles were in the austenite phase or a mixture of austenite and R-phase at 37 °C.

Conclusions The machining defects and structural imperfections of new Mani instruments are indicative of the difficulty in manufacturing nickel–titanium endodontic instruments. DSC measurements suggest that Mani instruments are capable of superelastic behaviour under clinical conditions.

Keywords: differential scanning calorimetry, endodontics, Mani NRT files, nickel–titanium, root canal, sterilization.

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Introduction

Rotary nickel–titanium (NiTi) instruments have become popular because of their substantially increased flexibility, their wider elastic limits and their superior resistance to bending and torsional failure compared to stainless steel instruments (Walia *et al.* 1988, 1989, Serene *et al.* 1995, Tepel *et al.* 1997, Torrisi 1999). NiTi instruments are considered suitable for negotiating curved root canals and have been shown to reduce the risk of transportation, zipping, stripping or ledging the canal (Esposito & Cunningham 1995, Glosson *et al.* 1995, Pettiette *et al.* 1999, Peters *et al.* 2001a,b). The use of these instruments in rotary motion, offers the possibility for more effective and predictable root canal preparation (Bentkover & Wenckus 1994, Himel *et al.* 1995, Gambill *et al.* 1996).

Endodontic instruments come into contact with saliva, blood and infected pulp tissue. As the instru-

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ments are frequently reused, it is essential they are sterilized after each use, to avoid cross-infection between patients. However, there is uncertainty regarding the potential changes to the physical and mechanical properties of NiTi instruments that are subjected to repeated sterilization cycles under autoclaves or dry heat sterilizers. Some authors reported an increase in the torque resistance of NiTi instruments (Kapila *et al.* 1992, Serene *et al.* 1995, Craveiro de Melo *et al.* 2002), others reported no consistent effect on bending and torsional properties (Silvaggio & Hicks 1997, Canalda-Sahli *et al.* 1998). Mize *et al.* (1998) concluded that heat sterilization had no effect on prolonging the clinical life of NiTi instruments.

Various studies have noted an increase in cutting efficiency of NiTi instruments following sterilization, possibly as a result of the increased surface microhardness that heat sterilization produces (Serene *et al.* 1995, Craveiro de Melo *et al.* 2002). However, other studies (Rapisarda *et al.* 1999, Schäfer 2002) have demonstrated a decrease in cutting efficiency of NiTi instruments, possibly caused by alterations in the surface of the instruments. It has also been observed that sterilization produced an increase in titanium oxide in the surface layer of the instruments (Rapisarda *et al.* 1999, Thierry *et al.* 2000a).

The nickel–titanium alloys used for endodontic instruments are based upon the equiatomic intermetallic compound NiTi, containing approximately 55% Ni and 45% Ti (Thompson 2000, Brantley 2001) and are similar to alloys originally used for orthodontics (Andreasen & Morrow 1978, Walia *et al.* 1988, Khier *et al.* 1991). These alloys exhibit two unique features that are of relevance to clinical dentistry: shape memory and superelasticity, as a result of reversible transformation of the austenite (parent phase) to the martensite (daughter phase) in the NiTi alloy (Civjan *et al.* 1975, Duerig & Pelton 1994, Thompson 2000). Martensitic transformation occurs as a function of temperature (temperature-induced martensite) or stress (stress-induced martensite). In the case of temperature-induced martensite, when the alloy is cooled through a critical transformation range (TTR), the crystal structure changes from a stable, cubic lattice, which is called austenite, to a monoclinic structure, known as martensite. The phenomenon starts at temperature M_s (martensite start) and ends at temperature M_f (martensite finish). It can be reversed by heating the alloy above the TTR, in the reverse transformation temperature range or RTTR (within austenite start temperature – A_s and austenite finish temperature – A_f), with the result that the properties of the alloy revert back to their previous higher temperature values, giving rise to the shape memory effect. Sometimes, the direct transformation from austenitic to martensitic NiTi includes an intermediate structure, called R-phase, which is a hexagonal lattice. In the case of stress-induced martensitic transformation, austenite is transformed to martensite as a result of the application of stress and reverts back to austenite when unloaded.

Machining characteristics and structural details that are created during the manufacturing procedures of endodontic instruments are essential to their clinical performance. The structure of the NiTi alloys is conveniently studied by differential scanning calorimetry (DSC), a method that allows the identification of NiTi phase (martensitic NiTi, R-phase or austenitic NiTi) at a given temperature, while temperature ranges and enthalpy changes for the phase transformations are also provided.

The purpose of this study was to study the effect of dry heat sterilization on surface characteristics and microstructure of 'as-received' Mani NRT 0.04 rotary instruments, using scanning electron microscopy (SEM) and DSC.

Material and methods

A total of thirty-three new Mani NRT instruments (MANI Inc, Toshigi-Ken, Japan), with a 0.04 taper, size 30 and 25 mm long, from the same batch (lot no. 5040679000) were examined. The sterilization cycles were conducted in a dry heat sterilizer (Technomedica, Milano, Italy), at 180 °C for 120 min. Samples were allowed to cool to room temperature for at least 30 min between cycles.

SEM

Twenty-seven instruments were examined in a type JSM840A (JEOL, Tokyo, Japan) Scanning Electron Microscope for surface characterization and potential alterations caused by the number of sterilization cycles. These specimens were divided into three groups. The first subgroup of three instruments, were examined for surface detail in the 'as-received' condition and after being subjected to 11 sterilization cycles. In the second group, 12 instruments were cross-sectioned and examined in subgroups of three instruments, after either 0, 1, 6 or 11 cycles of sterilization. Finally, in a third group, 12 instruments were sectioned longitudinally and studied in sub-

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Level	0	1	2	3
Surface roughness profile	Absence of roughness	Minimal presence of roughness	Moderate presence of roughness	Increased roughness

Table 1 Scale of values for the surface roughness profile of the Mani NRT specimens

groups of three instruments, after 0, 1, 6 or 11 sterilization cycles.

For cross- and longitudinal sections, samples were embedded in epoxy resin and after wet grinding with SiC papers, they were polished with diamond powder down to 1 μm with a grinding polishing machine (Struers A/S, Ballerup, Denmark). Then, the specimens were cleaned in an ultrasonic water bath for 15 min. After the sterilization cycles, they were vacuum coated with a thin layer of conductive carbon prior to SEM evaluation.

Scoring system

The level of surface roughness of the specimens was rated and scored in a scale of four appearances, using a pre-defined system and selected SEM pictures (Table 1).

The data were subjected to square root transformation and analysed as a completely randomized design with three replications. The means of the specimens of the first subgroup were compared with the *t*-test at the 0.05% level of significance. Comparison of means of the specimens of the second and third subgroups was made with the least significant difference, which was also set at 0.05% ($LSD_{0.05}$).

DSC

For DSC measurements, the remaining six endodontic instruments were examined: three Mani instruments were analysed in the 'as-received' condition and other three after being subjected to 11 sterilization cycles. DSC measures accurately the difference in thermal power supplied to a test specimen and an inert control specimen heated at the same rate. Structural transformations within the matrix of NiTi alloys are revealed as endothermic peaks on the heating DSC curves and as exothermic peaks on the cooling DSC curves. The measurements were conducted with a DSC 141 device (Setaram, Lyon, France) over a temperature range from −130°to 150 °C, using a liquid nitrogen cooling accessory (Setaram) to achieve subambient temperatures. During the measurements, the DSC cell was purged with dry nitrogen. Temperature calibration of the DSC apparatus was performed with *n*-pentane, deionized water and indium. The linear heating or cooling rate was 10 °C min^{-1}. For each analysis, the specimen was first cooled from room temperature to −130 °C, then heated to 150 °C to obtain the heating DSC curve (first heating) and subsequently cooled from 150 °C back to −130 °C (first cooling). To test the reproducibility of the measurements, the same heating–cooling cycle was repeated a further two times. The test specimens were carefully sectioned with a water-cooled, slow-speed diamond saw to minimize mechanical stresses that might change the proportions of the austenitic and martensitic NiTi phases from those in the 'as-received' instruments. Each test specimen consisted of three segments of the same file, each approximately 4–5 mm in length. Specimens were placed in an aluminium crucible and an empty aluminium crucible was used as the inert control specimen. The weight of the samples was approximately 11 mg.

Means were compared with the *t*-test. The level of significance was set at 0.05%.

Results

SEM observations

Two of the three samples in the 'as-received' condition, had a rounded, noncutting tip (Fig. 1), whilst the third had a sharp tip (Fig. 2). Debris, pitting, metal strips and deep milling marks (Figs 1 and 2) were detected on all instruments. After the samples were subjected to 11 sterilization cycles, debris remained and surface roughness was increased from level 1 (Fig. 1) or level 2 (Fig. 2) up to level 3 (Fig. 3), accordingly to the pre-defined scale (Table 1). Comparison of means between specimens that were subjected to 0 and 11 sterilization cycles (1.33 and 3.0 respectively) revealed that the difference in surface roughness was statistically significant ($P = 0.05$).

Scanning electron microscopic observations of cross- and longitudinal sections, indicated the presence of inclusions and holes, randomly distributed in the matrix of the NiTi alloy of all instruments after 0, 1, 6 and 11 sterilization cycles (Figs 4 and 5). Increased roughness, from level 1 to 3, was observed in specimens that were exposed to repeated sterilization.

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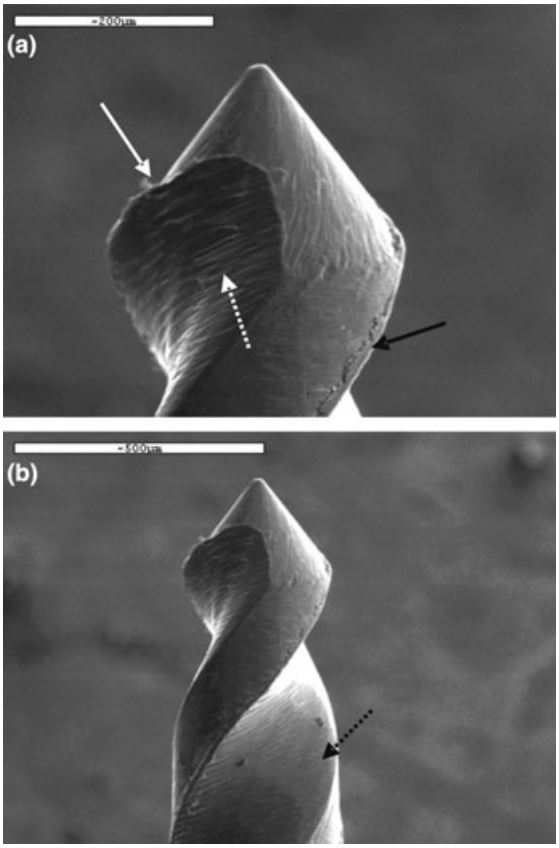

Figure 1 Mani instrument in the 'as-received' condition, with rounded, noncutting tip. (a) Magnification ×200, (b) magnification ×75. Presence of debris (white arrow), metal strips (white distinct arrow), machining defects along the cutting edges (black arrow) and pits (black distinct arrow). The surface roughness was considered minimal (level 1).

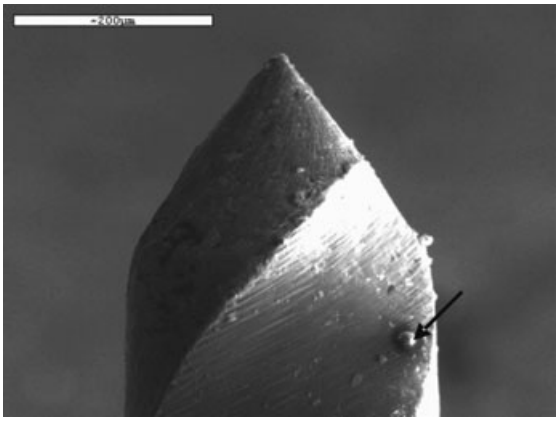

Figure 2 Mani instrument in the 'as-received' condition, with sharp cutting tip. Debris is detected near the cutting edge of the file (black arrow). Moderate roughness of the surface (level 2) was observed.

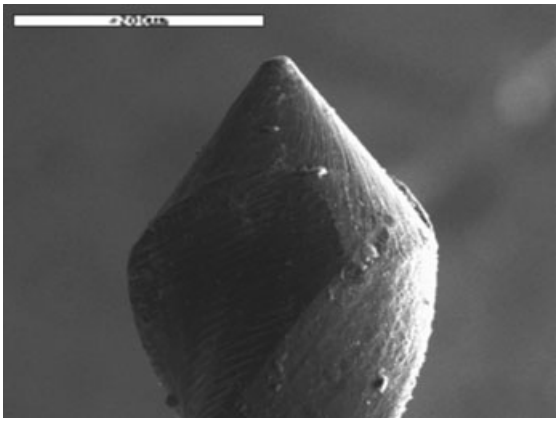

Figure 3 Mani instrument after 11 sterilization cycles. Increased surface roughness (level 3) was evident.

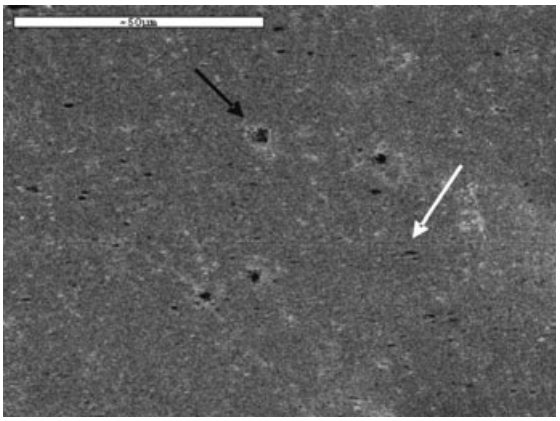

Figure 4 Surface (×1000) of longitudinal section of a Mani sample in the 'as-received' condition, with minimal roughness (level 1). Holes (black arrow) and inclusions (white arrow) randomly distributed within nickel–titanium matrix.

These findings were morphometric rather than alterations in chemical composition induced by sterilization procedures, as shown by the back-scattered electron images of the same regions (Fig. 6). The analysis of variance of the cross-sectioned specimens revealed nonsignificant differences in surface roughness between 0 and 1 sterilization cycles as well as between 6 and 11 sterilization cycles. However, the differences between 0 or 1 and 6 or 11 sterilization cycles were statistically significant (Table 2). For longitudinal sections, analysis of variance showed significant increases in surface roughness between all specimens that were subjected to different number of sterilization cycles, with exception of the specimens that were subjected to 0 and 1 sterilization cycles (Table 2).

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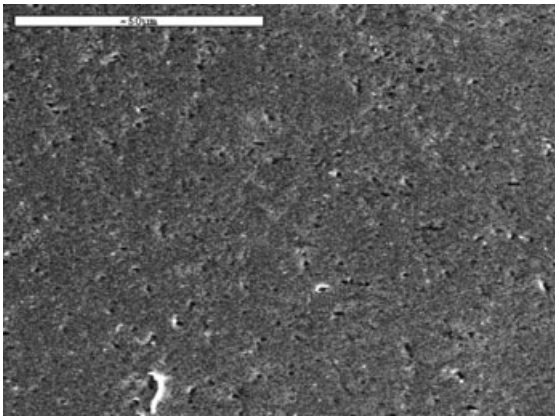

Figure 5 Surface (×1000) of longitudinal section of a Mani sample that was subjected to 11 sterilizations. Increased roughness of the surface (level 3) was the prevalent finding.

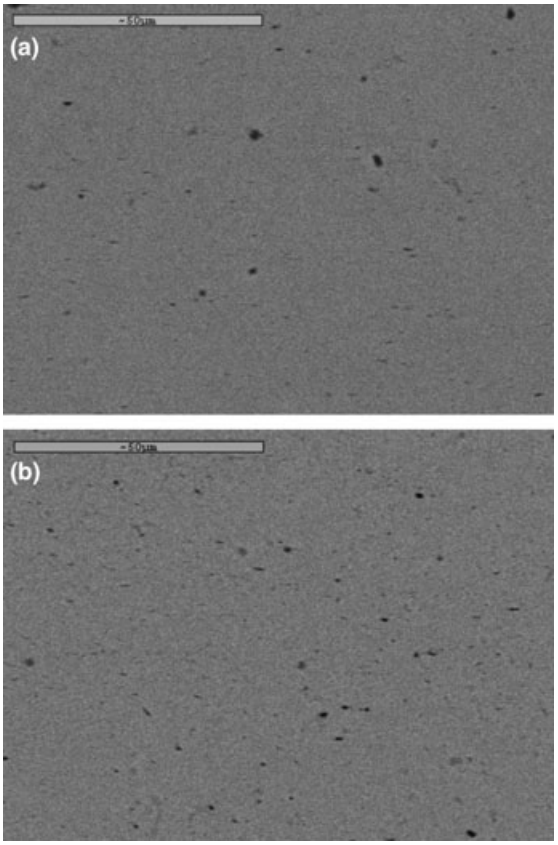

Figure 6 Figures (a) and (b) showing the back-scattered electron microphotographs of the regions shown in Figs 4 and 5 respectively.

Table 2 Comparison of mean values for the surface roughness level between specimens that were subjected to different number of sterilization cycles

Mean value of surface roughness level	Number of sterilization cycles				
	0	1	6	11	$LSD_{0.05}$
Cross-sectioned specimens	1.0	1.3	2.3	3.0	0.25
Longitudinal sectioned specimens	1.0	1.0	2.3	3.0	0.16

$LSD_{0.05}$, least significant difference at 0.05% level of significance.

DSC measurements

Figure 7 shows characteristic DSC plots for heating and cooling curves, following subtraction of the baseline, for one of the 'as-received' specimens. The DSC heating curve shows a two-step endothermic transformation with overlapping peaks. These peaks correspond to the initial transformation of the martensite (M) to R-phase (R) at lower temperatures, followed by transformation of R-phase to austenite (A) at higher temperatures. The DSC exothermic cooling curve shows a one-step distinct transformation. The transformation product of this peak most likely corresponds to the transformation from austenite (A) to R-phase (R), whereas the transformation from R-phase to martensite (M) at lower temperatures is difficult to identify, as it is dissimulated in the baseline of the thermogram. At a peak temperature of approximately 18.3 °C, 50% of the martensitic phase has been transformed to R-phase upon heating (R_p temperature), while at a peak temperature of approximately 33.8 °C, 50% of the austenite phase was present (A_p temperature). The transformation to the

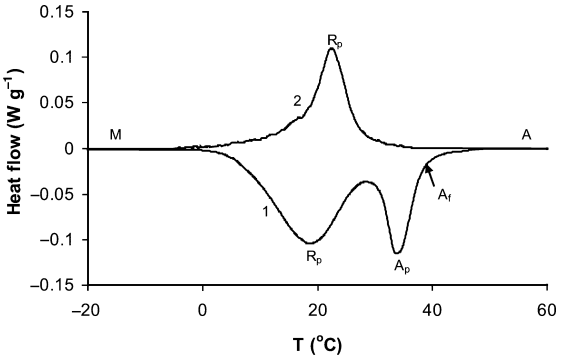

Figure 7 Differential scanning calorimetry thermogram for the 'as-received' sample. 1, heating; 2, cooling.

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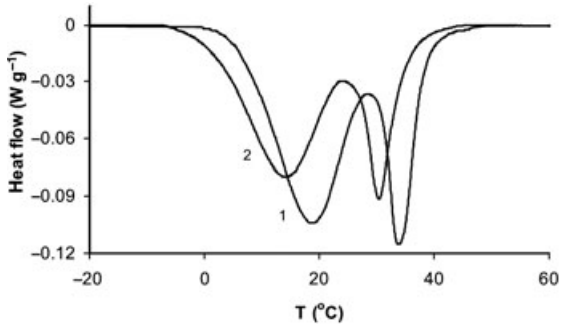

Figure 8 Differential scanning calorimetry thermogram during heating. 1, 'as-received' sample; 2, sample after 11 sterilization cycles.

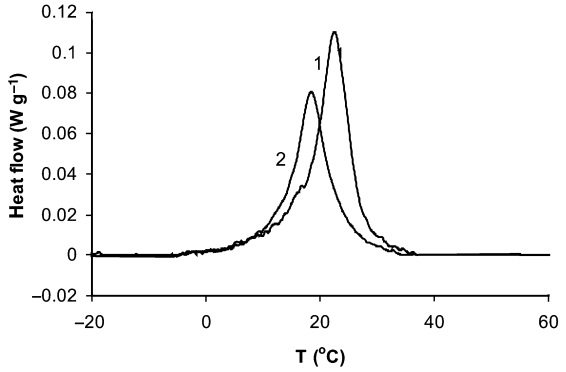

Figure 9 Differential scanning calorimetry thermogram during cooling. 1, 'as-received' sample; 2, sample after 11 sterilization cycles.

austenitic NiTi phase was completed at approximately 38.5 °C (A_f temperature). The area under the peaks represents total enthalpy change (ΔH) of approximately 13.5 J g^{-1}, for the overall transformation from the martensitic to austenitic NiTi phase. The DSC exothermic cooling curve shows a one-step distinct transformation. The transformation product of this peak most likely corresponds to the transformation from austenite (A) to R-phase (R), with R_p temperature during cooling at approximately 22.4 °C, whereas the transformation from R-phase to martensite (M) at lower temperatures is difficult to identify and is dissimulated in the baseline of the thermogram. The enthalpy change during cooling of the sample is approximately 5.1 J g^{-1}.

In the specimens that were subjected to 11 sterilization cycles the heating curve also shows two endothermic, overlapped peaks, while in the cooling curve one exothermic peak is revealed.

In Figs 8 and 9, heating and cooling curves of representative specimens in the 'as-received' condition and after 11 sterilization cycles are presented. Peak temperatures on the heating curve of specimens that underwent 11 sterilizations were R_p *c.* 14.1 °C and A_p *c.* 30.3 °C, with the austenite finish temperature A_f *c.* 34.9 °C. The main peak in the cooling curve most likely corresponded to the initial transformation of the austenitic phase to R-phase, with peak temperature R_p *c.* 18.4 °C. The enthalpy changes were 11.3 J g^{-1} during heating and 4.5 J g^{-1} during cooling.

Table 3 summarizes the results from the DSC evaluations of all Mani instruments examined in

Table 3 Values obtained from differential scanning calorimetric analyses for the 'as-received' and sterilized Mani NRT specimens

Traits	'As-received' samples	Samples after 11 sterilizations	Difference
R_p (peak temperature	15.6	14.1	
of martensite to R-phase)	17.2	15.8	
on heating (°C)	18.3	18.4	
Mean R_p (on heating)	17.03	16.10	NS
A_p (peak temperature of	27.9	28.2	
R-phase to austenite)	29.9	28.4	
on heating (°C)	33.8	30.3	
Mean A_p	30.53	28.97	NS
A_f (austenite finish	33.5	33.2	
temperature) on	36.1	32.5	
heating (°C)	38.5	34.9	
Mean A_f	36.03	33.53	NS
R_p (peak temperature	17.5	18.0	
of austenite to R-phase)	19.5	18.2	
on cooling (°C)	22.4	18.4	
Mean R_p (on cooling)	19.80	18.20	NS

NS, statistically nonsignificant difference at $P = 0.05$.

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the 'as-received' condition and after 11 sterilization cycles. Comparison of the means revealed nonsignificant difference between specimens in the 'as-received' condition and specimens that were subjected to 11 sterilization cycles, for any of the four traits.

Discussion

Scanning electron microscopic observations of new Mani NRT rotary NiTi instruments showed irregularities and structural defects in all samples. In the outer surface of the instruments debris, pitting and metal strips were detected, while inclusions and holes in the NiTi matrix were revealed in cross- and longitudinal sections of the samples. The presence of the adherent material probably occurred from decomposition and oxidation of the lubricating oil used in cutting and machining the instrument during manufacture (Thompson 2000, Martins *et al.* 2002). Inclusions are presumed to be primarily titanium carbonitrides, with a few nickel–titanium oxides, which are formed during vacuum melting in graphite crucibles during the manufacture of NiTi alloys, while holes possibly originate during the wire-shaping process (Lee *et al.* 1988, Melton 1990, Thompson 2000). Morphometric variations amongst the instruments of the same batch can be attributed to the thermomechanical history of each instrument during its manufacturing procedure. These findings are in agreement with the findings of other authors that reported structural defects in new Lightspeed (Marsicovetere *et al.* 1996, Eggert *et al.* 1999, Alapati *et al.* 2003) and new ProFile (Martins *et al.* 2002, Alapati *et al.* 2003) NiTi endodontic instruments and are indicative of the difficulty in manufacturing defect-free NiTi instruments, because of the complex process of NiTi alloy production and the difficult machining operations that manufacture of NiTi endodontic files requires (Thompson 2000).

The consequences of these imperfections in terms of durability and cutting efficiency in clinical use of the instruments are difficult to assess and have not yet been adequately evaluated. According to Schäfer (1997) and Thompson (2000) the presence of these defects on the cutting edges of the instruments, is responsible for their relatively low cutting efficiency and may compromise their corrosion resistance. On the other hand, Eggert *et al.* (1999) claimed that the presence of these imperfections was probably not relevant to their clinical performance.

In all samples that were subjected to repeated sterilization cycles, an increased surface roughness occurred compared to those that were not sterilized. A possible explanation might be an alteration in thickness of the passive, oxide layer that covers NiTi surfaces. Rapisarda *et al.* (1999) and Thierry *et al.* (2000a,b) found an increase in the TiO_2 oxide surface layer of NiTi alloys that were exposed to sterilization.

Differential scanning calorimetry has been used extensively for characterization of NiTi orthodontic wires (Lee *et al.* 1988, Bradley *et al.* 1996, Brantley 2001, Iijima *et al.* 2002) and lately to investigate phase relationships within NiTi matrices of popular endodontic instruments: ProFile, Lightspeed, Quantec and Hero in the as-received condition and after clinical or simulated clinical use (Torrisi 1999, Brantley *et al.* 2002a,b, Kuhn & Jordan 2002). In these studies, substantial variability occurred in values (Table 2) between specimens from different brands as well as between specimens from the same brand. This is attributed to differences in the starting wire blanks and perhaps processing procedures for the batches of each instrument product. Mani NRT endodontic instruments have been introduced relatively recently to endodontics, therefore no information exists in the literature.

As shown in Fig. 7, the phase transformation within NiTi alloy of the instruments is reversible. The reversibility is not affected after repeated heating and cooling in the same instrument or after 11 sterilization cycles.

According to Duerig & Pelton (1994), superelasticity is the enhanced elasticity that occurs when unloading between A_s and M_d temperature (defined as the temperature above which stress-induced martensite can no longer be formed and assumed to be 25–50 °C higher than A_f). If austenite is not present, transformation cannot take place and superelasticity does not exist. DSC measurements showed that at a temperature of approximately 37 °C, which corresponds to the temperature of the oral environment, new and sterilized specimens are completely austenite or a mixture of austenite and R-phase. This means that these instruments could undergo superelastic behaviour during use in the root canal, where the imposition of stress up to 8% strain (Thompson 2000) causes transformation to the martensitic NiTi phase. When the instrument is unloaded, the alloy reverts back to the original austenitic phase. Therefore, the examined specimens can be considered capable of superelastic behaviour under clinical conditions.

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Table 3, shows the variability of values determined from DSC studies for the Mani NRT samples. From these values, no trend is obvious for the possible shift of the heating and cooling curves for the instruments that were subjected to 11 sterilization cycles compared with specimens in the 'as-received' condition.

The enthalpy changes observed for the overall martensitic NiTi to austenitic NiTi transformations during heating ranged from 13.5 J g^{-1} for the 'as-received' specimens to 11.3 J g^{-1} for the specimens after 11 sterilization cycles. The changes in the transformation from austenitic to R-phase during cooling ranged from 5.1 to 4.3 J g^{-1}. These values are lower than the ones during heating, probably because during cooling the transformation from R-phase to martensitic NiTi was not observed. The calculated values of the enthalpies lie within the range of 1.7–19.2 J g^{-1}, which has been reported for some nickel–titanium orthodontic wires (Bradley *et al.* 1996, Brantley 2001). The relatively high enthalpy changes of the Mani instruments examined, for the overall transformation during heating, are possibly due to low amounts of stable, work-hardened martensitic NiTi (which does not undergo transformation to austenitic NiTi) in the instrument after manufacturing. This also may be the cause for the relatively lower values that were observed at samples that were subjected to 11 sterilization cycles and might suggest that instruments that undergo multiple sterilizations present a less advantageous microstructure. Future research is required to verify this hypothesis.

Conclusions

Machining defects and structural imperfections of new Mani instruments are indicative of the difficulty in manufacturing defect-free Ni–Ti endodontic instruments. DSC measurements of all instruments show that phase transformation within the NiTi matrix is reversible. The reversibility is not affected by repeated heating and cooling cycles, as assessed by DSC measurements. At the temperature of the oral environment, the specimens are completely austenite or a mixture of austenite and R-phase, suggesting that they can be considered capable of superelastic behaviour during clinical use.

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