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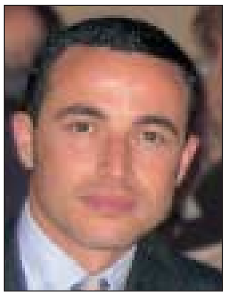
Surface properties of nickel-titanium rotary instruments



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NiTi rotary systems are able to prepare root canals to excellent taper, with less canal transportation, greater preservation of tooth structure, and allow faster canal preparation than hand instruments. Since the capability of NiTi instruments to efficiently enlarge root canals depends on their surface properties, surface treatments of NiTi instruments are examined and the effects of NaOCl, sterilisation processes and electropolishing are discussed.

Introduction

In 1988, Walia et al[1] introduced a new alloy for the manufacturing of endodontic instruments: nickel-titanium (NiTi). Several studies have shown that NiTi rotary systems are able to prepare root canals with excellent taper, less canal transportation, greater preservation of tooth structure, and much faster than hand instruments[2,3]. NiTi files exhibit more elastic flexibility in bending and torsion, as well as superior resistance to corrosion compared with stainless steel files[4,5]. Over time, an increasing number of new NiTi rotary systems have been developed with files possessing unique design features in terms of cross-sectional shape, taper, surface treatment or number and angle of cutting flutes. However, despite the evident advantages of these new systems, NiTi rotary instruments may experience failure due to fatigue or torsion[6,7].

Fracture due to torsion occurs when the tip or any other part of the instrument binds in the canal whilst still rotating. When this occurs, the elastic limit of the metal is exceeded and fracture of the instrument becomes inevitable. This type of fracture has been associated with the application of excessive apical forces during instrumentation[8].

Pruett et al[9] suggested that flexural fatigue plays an important role in the fracture of NiTi instruments. Cyclic fatigue is caused by repeated tension-compression stresses. Rotation subjects an instrument to both tension and compression stresses in the area of the curvature. The repeated tension-compression cycle, caused by curved canals, increases cyclic fatigue of the instrument over time and may be the most important factor in instrument separation[10].

Scanning electron microscopic studies showed the presence of micro-fissures and defects on the surface of both new and used NiTi instruments. These could be the prelude to an imminent, further deterioration of the instrument and could explain the reported increased breakage of these instruments under normal operative conditions[11]. According to Kuhn at al[12], during the breakage process, the 'crack-nucleation' stage is facilitated by a high density of surface defects, and therefore the fatigue failure is largely a crack-propagation process. Applying thermal treatment (recovery) of NiTi instruments in order to decrease the work-hardening of the alloy could be used to improve the longevity[13].

To better understand the fracture of NiTi instruments in root canals and in operative conditions that are apparently simple, alternative mechanisms were proposed, such as corrosion failure[14,15]. In particular, the effect of sterilisation processes, NaOCl corrosion and electropolishing techniques might have an influence on the susceptibility to fracture of NiTi instruments.

Surface properties of NiTi instruments

Since surface characteristics affect the instruments, the capacity of a file to enlarge a canal efficiently is dependent on its surface properties. Several constituents can be found on the surface of NiTi instruments. In fact, along with Ti and Ni, C, O and other components (Si, Ca, Mg) were observed due to environmental contamination[16] (Fig 1). Since the surface of the NiTi instrument is in contact with air containing oxygen and carbon, the concentration of nickel and titanium increases from the surface towards the core. A typical surface analysis of NiTi instruments is: C (62%), O (30%), Ni (2%) and Ti (5%) (Fig 2). A Ni/Ti surface ratio of 0.30 was observed with NiTi instruments.

Martins et al[17] reported adherent deposits containing carbon and sulphur, which appeared to originate from the lubricating oil used during the production process (Fig 3). These deposits served as sites for subsequent accumulation of debris during clinical use and could be an adverse factor, increasing the friction between a NiTi instrument and dentine walls. Moreover, it is assumed that the presence of surface irregularities on NiTi files may compromise their cutting efficiency and make them more prone to corrosion and fractures[18]. During the grinding of the cutting edges, small scratches and grooves are created on

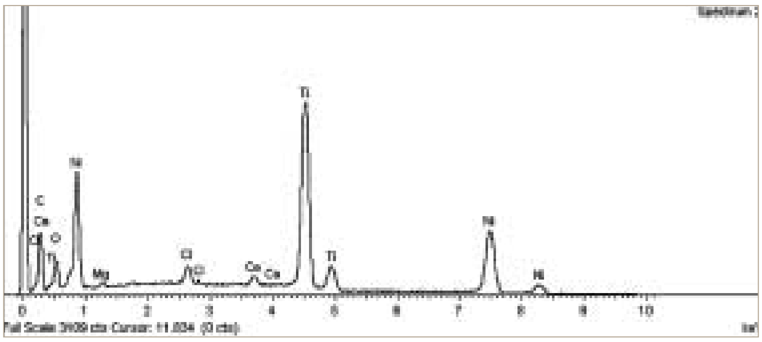

Fig 1 Contaminants usually found on the surface of NiTi instruments.

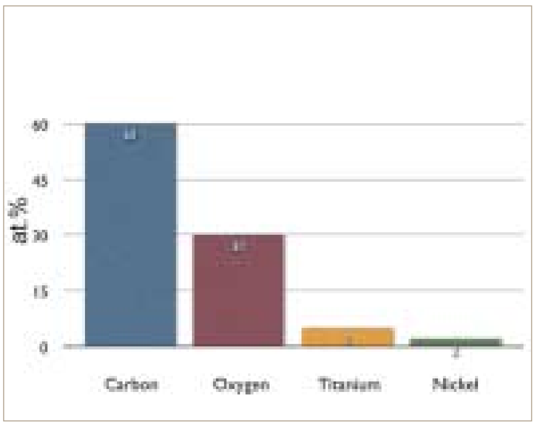

Fig 2 Surface analysis of NiTi instruments: given are the atomic ratios (%) of the different elements.

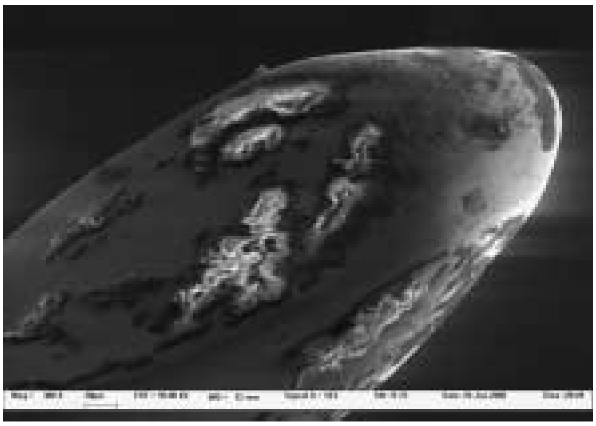

Fig 3 Adherent deposits on the surface of a new NiTi instrument.

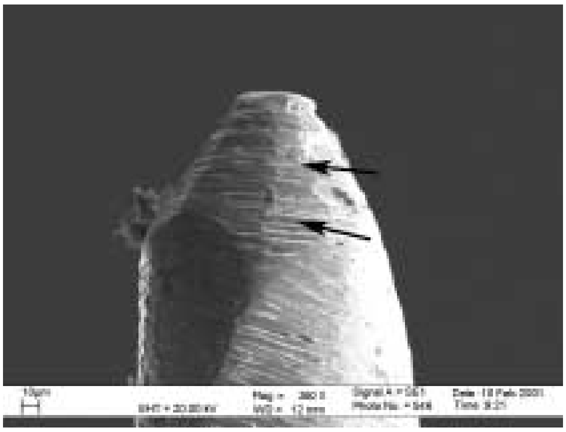

Fig 4 Presence of metallurgic defects (micro-cracks, machining marks and metal flash) on the surface of a new NiTi instrument (indicated by arrows).

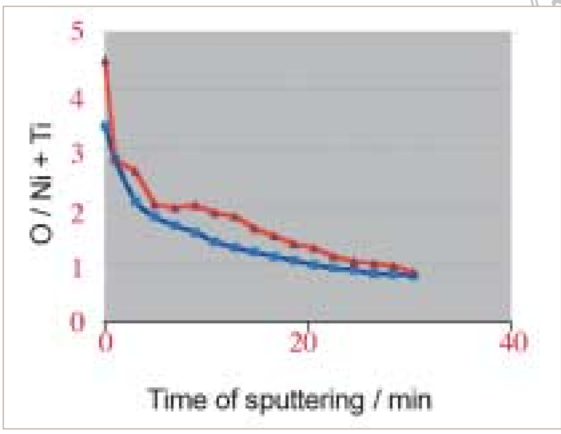

Fig 5 An increased amount of oxygen was observed on the surface of NiTi instruments after autoclave sterilisation (red line) compared with new instruments (blue line). The time of sputtering relates to the sampling depth of the surface of the instruments.

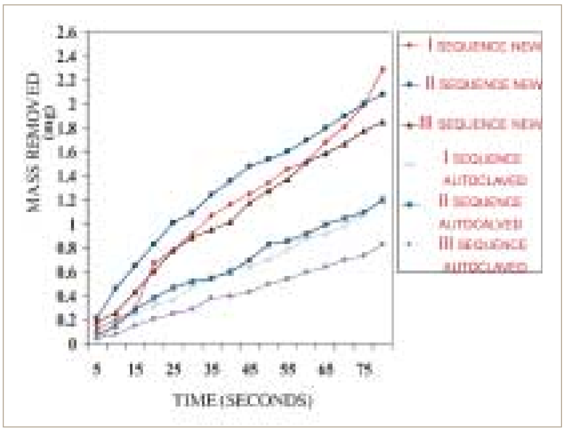

Fig 6 Decreased cutting efficiency of NiTi instruments after sterilisation processes. Unsterilised and sterilised NiTi instruments were used under identical conditions, on resin surfaces, and the removed mass of resin recorded. The sharper the cutting edges of the instruments, the greater was the material removal. Obviously, even if they were used for two or three sequences, unsterilised new instruments removed a markedly greater mass compared with sterilised instruments. The cutting efficiency of sterilised instruments decreases with an increasing number of sterilisation cycles.

the surface of NiTi instruments[19,20] (Fig 4). For this reason, many authors suggest that new rotary NiTi instruments should be cleaned with ultrasound before clinical use and sterilised in order to eliminate any metal residue and surface debris[21–23].

Effect of sterilisation on NiTi instruments

Infection control guidelines require the sterilisation of instruments that come in contact with tissues or blood[24]. For this reason, CDC (Centers for Disease Control and Prevention) guidelines recommended that endodontic files should be sterilised using heat, for example autoclaving[25]. Autoclaving of NiTi endodontic instruments is necessary both before and after use to remove all biological contaminants[26].

Using a spectroscope, Shabalovskaya and Anderegg[27] examined NiTi alloy surfaces after repeated sterilisation cycles. They noticed that autoclaving at 120°C and 21 psi resulted in an alteration of the concentrations of Ni, Ti, O and C on the alloy surface. The extent of the changes was proportional to the duration of sterilisation. A decrease in Ni concentration was also found on the surface of the instruments with increasing time (1–2 hours in the autoclave). Therefore, it was suspected that saturated steam in the autoclave causes oxidation on the files[27]. After 11 sterilisation cycles, debris was detected on the surface of NiTi instruments, while surface roughness increased markedly[28].

Autoclaving, especially when repeated, increases the quantity of oxygen present on the surface of NiTi instruments (Fig 5) and creates conditions that reduce their cutting efficiency (Fig 6)[16]. According to these studies, repeated autoclave sterilisation of endodontic NiTi instruments has been shown to adversely affect the cutting efficiency of these instruments (Fig 6). In particular, files that underwent five cycles of sterilisation experienced a reduction in cutting ability of approximately 16.1% compared with untreated files. After 10 cycles of sterilisation, the cutting efficiency was further reduced by up to 50.8%[13]. The explanation for these undesirable effects of sterilisation may be that sterilised instruments undergo changes on their outer surfaces and in the chemical composition of the surface layer. It was observed that sterilisation produced an increase in titanium oxide (TiO_2) in the near-surface layer of the instruments[16].

Effects of NaOCl on NiTi instruments

Sodium hypochlorite is the most commonly used irrigant in endodontics (see corresponding article in this issue of the journal) and can also be used to clean root canal instruments. NaOCl is corrosive to many metals and selectively removes Ni from NiTi alloys[29]. NaOCl contains active ClO^- ions, and it is well known that ClO^- is an aggressive ion, which generally increases corrosion rates[30]. NaOCl could lead to micro-pitting if used as a disinfectant when cleaning NiTi instruments. Many studies have investigated the effect of NaOCl on the mechanical properties of NiTi instruments. According to these studies, NaOCl treatment did not alter the cutting efficiency[31], did not lead to a significant reduction in flexural or torsional strength of NiTi instruments (after 10 cycles of cleaning, 2.5 h total immersion time) with a NaOCl solution and multiple clinical use (including exposure to 2.5% NaOCl as an irrigating solution for an unspecified period). It also did not adversely affect the cyclic fatigue of the NiTi instruments[32]. On the other hand, Stokes et al[29] described pitting corrosion of the file surface after 1 h of immersion in 5.25% NaOCl and speculated that manufacturing factors affected the corrosion of these instruments (Fig 7).

Although there are no reports in the literature about corrosion failure of files, it is likely that pitting or crevice corrosion might occur first and then promote fatigue failure, altering the fracture mechanism from conventional fatigue failure to corrosion failure.

Although NaOCl is commonly proposed for cleaning[21], O'Hoy et al[15] reported corrosion of NiTi instruments after immersion in a NaOCl solution overnight. This is in agreement with the observations of Stokes et al[29], in as far as pitting corrosion of NiTi surfaces was observed after 1 h of immersion in 5.25% NaOCl.

In a recent study (Bonaccorso et al, unpublished results), the presence of deposits on the flutes and the tip of NiTi instruments (Fig 8) were examined after NaOCl treatment. Due to different metals (NiTi and stainless steel) present in the surface of the NiTi instruments (in this study RaCe rotary instruments were evaluated) galvanic reactions and corrosion processes may be caused by the presence of an

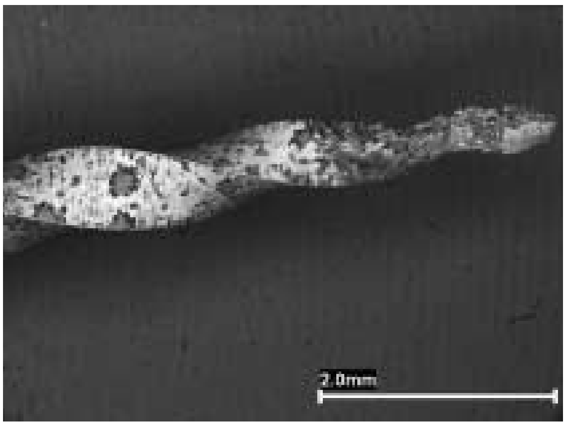

Fig 7 Typical pitting corrosion on the surface of a used NiTi instrument.

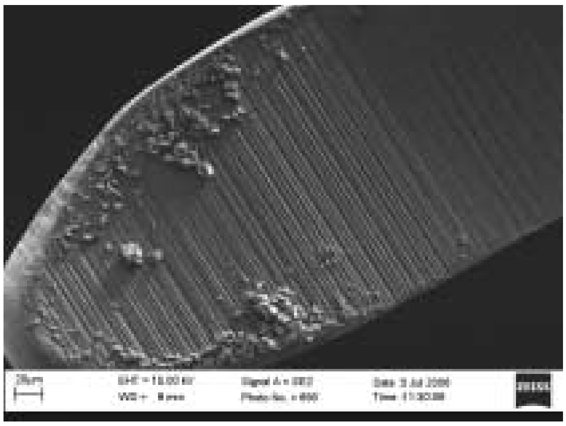

Fig 8 Presence of deposits after NaOCl treatment on the surface of a NiTi instrument.

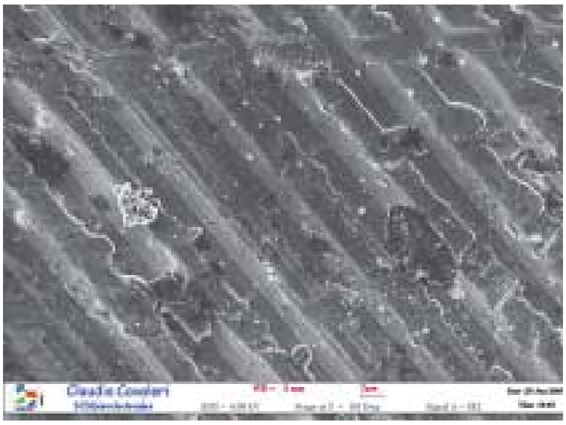

Fig 9 Magnification of the surface deposits on defects of a NiTi instrument after NaOCl treatment .

Fig 10 Presence of Fe deposits on the tip of a NiTi instrument after NaOCl treatment.

electrolytic solution such as NaOCl. In addition, NaOCl solutions initiated a widespread chemical oxidation on the surface of the instruments. In the case of NiTi instruments, the oxidation process could not be accurately assessed since oxide constituents were already present on the surface. However, these instruments an increased quantity of depositions (NaOCl residues, oxides and metal deposits) was observed in the surface defects (milling marks, groves and micro cracks) (Fig 9). Therefore, it seems that NaOCl more easily corrodes the minor surface defects already existing on the surface of non-electropolished instruments (see below) and may thereby weaken the instruments. It is worth mentioning that in this study, pitting and corrosion occurred on the shaft of the instruments, while no marked corrosion was observed on the flutes or near the tip of the instruments. At first sight, these results seem to disagree with the observations of other studies evaluating corrosion processes on ProTaper and GT Rotary NiTi instruments[15,33]. However, after careful analysis these differences can be explained as follows: ProTaper and GT Rotary NiTi instruments have shafts coated with gold, which possess a high redox potential ($E°_{Au3+/Au}$=+1.50 V). This property causes the shaft to behave as a cathode, and the NiTi cutting part ($E°_{Ni2+/Ni}$= -0.25V and $E°_{Ti2+Ti}$= V) as the anode at which corrosion arises[34]. In contrast, the shaft of RaCe instruments is made of stainless steel in which iron ($E°_{Fe2+/Fe}$= -0.74V) possesses a lower redox potential than the cutting part made of NiTi. Therefore, the corrosion occurred on the shaft (the anode) and Fe is deposited on the NiTi alloy (Fig 10). In particular, larger amounts of deposit were observed at the tip region of the instruments, because it is known that the end of the tip induces a higher current density.

The different composition of the shaft and the flutes of rotary NiTi instruments obviously results in galvanic reactions that lead to shaft corrosion and metal deposits on the tip and flutes of the instruments. For this reason, a shaft made of NiTi alloy or plastic may be a solution to reducing the corrosive effects on these instruments when in contact with NaOCl solutions.

Electropolished surface treatment

Electropolishing is the controlled electrochemical removal of surface roughnesses, resulting in a shiny appearance and improved mechanical properties. Sometimes described as 'reverse plating' or 'super passivation', this process has a levelling effect that creates smoothness and increased reflectivity. More importantly, the deformed, amorphous outer layer of the metal is removed, leaving a surface free of embedded contaminants and work-induced residual stresses[34]. Generally, electropolishing is accomplished by connecting the metal part to be processed to the positive electrode (the anode) of a DC power supply. The part is then immersed in a heated electrolytic bath that contains metal plates connected to the negative electrode (the cathode). The electrical reaction causes ionic conduction resulting in the removal of metal particles from the anode. During the proce-

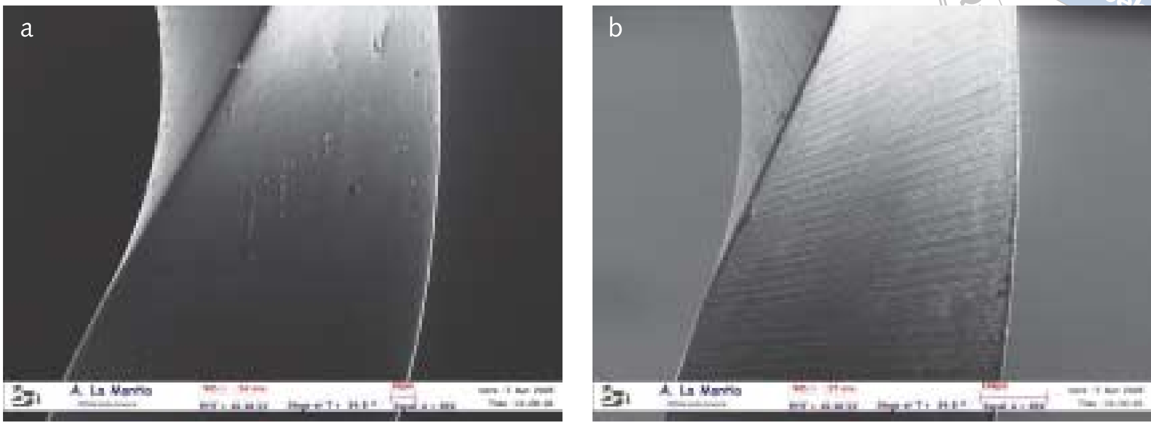

Fig 11 Scanning electron microscope image of RaCe instruments: (a) showing no surface defects due to electropolishing surface treatment; and b) with clearly visible surface defects without any surface treatment.

dure, the products of this anodic metal dissolution react with the electrolyte to form a surface film on the metal.

A recent study on NiTi instruments has shown that electropolishing surface treatment is able to remove machining grooves (Fig 11) and can improve the fracture-related fatigue resistance[18]. During machining operations, small scratches and grooves are invariably made on the endodontic instrument surface by the cutting tool. These milling marks can reduce fatigue resistance. In fact, a significantly lower number of manufacturing marks was observed for NiTi RaCe files after a process of electropolishing compared with untreated files, which showed significantly lower fatigue resistance despite the identical design and sizes. Since both the surface defects and internal stresses can act as adverse factors to the mobility of martensite interfaces, reducing the presence of machining marks with electro-polishing procedures improves the instrument's longevity.

■ Electropolishing and fatigue resistance

A recent study revealed that untreated NiTi RaCe instruments displayed similar fatigue resistance times compared with Hero and Mtwo files, but shorter times than ProFile and K3 files[18]. These findings strongly indicate that instruments having triangular cross sections with sharp cutting edges do not have the same flexibility as files with a U-shaped cross-

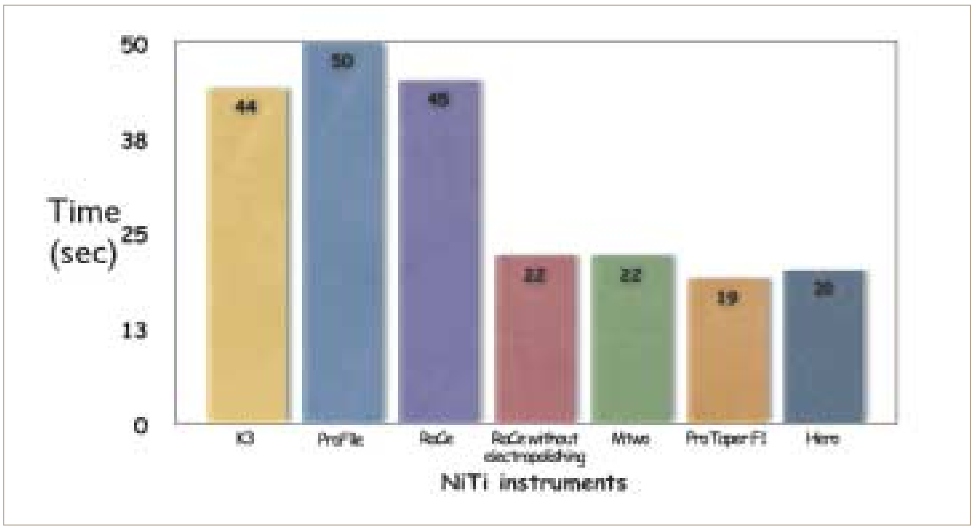

Fig 12 Fatigue resistance of different rotary NiTi instruments: mean values of fracture time (seconds) for each type of instrument are given.

sectional design. However, it should be remembered that the surface of the commercially available RaCe instruments is treated by an electropolishing procedure, which reduces the presence of machining damage on the file surface and increases the fatigue resistance to values comparable to ProFiles (Fig 12).

The fatigue resistance of root canal instruments influences the results of instrumentation in curved canals. In fact, repeated bending stresses in curved canals causes metal fatigue and subsequent instrument separation[35].

The clinical effectiveness of root canal instruments made of the same alloy and identical sizes mainly depends on their design features. The design of root canal instruments (cross-sectional geometry, size, taper, number and angle of flutes, surface treatment) seems to play an important role in fatigue resistance[36].

Conclusions

In current endodontic practice, it is generally accepted that rotary NiTi instruments are able to prepare even severely curved root canals. An increasing number of new NiTi rotary file systems are currently available. Recently developed NiTi files possess unique design properties in terms of cross-sectional shape, taper, surface treatment (electropolishing), number and angle of flutes etc. However, less is known about the possible damaging effects of cleaning procedures (NaOCl) and sterilisation processes, and positive effects of surface treatments. From this review the following can be stated:

- The surface of NiTi instruments contains high concentrations of C, O, and of environmental contaminants.
- The negative effects of repeated sterilisation processes are evident in terms of a significant decrease in cutting efficiency due to an increase of TiO in the surface layer.
- NaOCl treatment results in marked surface modifications of NiTi instruments due to galvanic reactions that lead to shaft corrosion and metal deposits on the tip and flutes of the instruments.
- Electropolishing results in distinct improvement of the surface composition and properties of NiTi instruments and produces a surface layer free of organic and inorganic contaminants.

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