Investigations of endodontic instruments in NiTi superelastic alloy

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Abstract

Endodontic instruments in NiTi alloy, useful in dentistry for restorative procedures, are investigated in terms of functionality, mechanical wear and temperature dependence. The instruments are studied by dynamical mechanical thermal analysis, differential scanning calorimetry, wear tests and scanning electron microscopy. The characterisation is given as a function of the temperature in the range between –30 °C and +80 °C. Obtained results point out the best conditions to use the files inside the dental root channels minimising the risk of their occasional rupture.

Riassunto

Strumenti endodontici in lega NiTi, utilizzati in Odontoiatria per procedure ricostruttive, sono investigati in termini di funzionalità, usura meccanica e dipendenza dalla temperatura. Gli strumenti sono studiati adoperando analisi termo-meccaniche, calorimetria a scansione differenziale, prove di usura e microscopia elettronica a scansione. La loro caratterizzazione è data in funzione della temperatura nel range -30°C e +80 °C. I risultati ottenuti indicano le condizioni migliori per poter usare le lime all'interno dei canali della radice del dente minimizzando il rischio della loro rottura occasionale.

INTRODUCTION

The NiTi alloy has several applications in medical field: orthopaedic clamps, dental implants, surgical staples, endoscopic instruments, cranial plates and cardiological guidewires represent some of applied devices. Special devices are employed in dentistry such as orthodontic wires, useful to correct the dental position, palatal expanders and endodontic instruments. These lasts, object of this investigation, are used to prepare the tooth canal for restorative procedures. They are little conical files, which are employed to erode the canal and to enlarge its diameter maintaining the original shape [1].

It's well known that the NiTi alloys show a crystalline transition from austenite to martensite phase and vice versa when they are submitted to cooling or heating processes, respectively. This transformation occurs in a narrow temperature interval of about 20 °C, which can be shifted from about –100 °C to 100 °C changing the NiTi composition.

The austenitic arrangement is stable at high temperature and shows a face centred cubic structure with unit cell angle of 90°. The martensitic phase, unstable at high temperature, has a lower rigidity and presents a deformed structure, consisting in a cubic structure with an angle of 96°. These materials show "shape memory" behaviour when are mechanically deformed at low temperature, in martensitic phase, and successively heated up to the austenitic phase. Furthermore, the NiTi alloys present "superelastic" properties if deformed in the austenitic phase within a definite temperature range [2].

The good biocompatibility and the high corrosion resistance have made these NiTi alloys excellent for special medical applications. In the alloy the Ni is chemically joined to the Ti in a strong intermetallic bond and the alloy surface shows a thin TiO_2 layer which acts as a barrier for the Ni release, so the risk of reaction, even for patients with Ni-sensitivity, is extremely low [3].

The upper described peculiarities have suggested substituting, in special applications, the traditional stainless steel devices with NiTi alloys. Tab. I show a comparison between the some physical properties of the NiTi alloy and those of classical stainless steel. The alloy has higher elasticity, ultimate tensile stress (UTS) and biocompatibility than to the stainless steel [4].

In dentistry field, the instrumentation used to prepare the dental root channels to successive restoration procedures represents an interesting aspect of NiTi application. The endodontic files must be highly flexible to penetrate in the bent channels and their erosion must respect the anatomic shape of the channel and must be sufficiently resistant to avoid the file rupture inside the tooth. The clinical use of NiTi based files has conducts at secure advantages for the endodontic therapy [5].

A stainless steel file inserted into a bent channel is elastically deformed by high stress and produces a non-uniform pressure on the canal walls, with high risk to modify the original canal shape during its rotation. The NiTi file, instead, easily follows elastically the shape of the dental canal producing, during its rotation, a homogeneous action of erosion of the canal walls and driving out the produced debris tanks to its screw helicoidal shape.

The final size of the channel increases proportionally with the file diameter, especially for stainless steel files with section higher than 3 mm. The NiTi instruments, instead, permit to increase the dimension of the treated canal with a higher control of its diameter without formation of wall steps and of lateral perforations. Moreover, the NiTi with different conicity permits to obtain a canal preparation "crown-down" type [5].

The NiTi file shapes are different from traditional stainless steel ones. Their particular spiral screw permits to reduce the dangers of accidental microfractures formation in the tooth and the deformation of the cutting blade. During the file rotation the pressure of the blade against the dentinal walls is almost constant and the abrasion is uniform. Many instruments have a sharp apical tip, to prevent the formation of wall steps, permitting an easily guide of the device into the dental channel with a low probability of tip rupture [6].

TABLE 1 - Comparison between some physical properties of NiTi alloy and Stainless Steel

Physical Property	NiTi	Stainless Steel
Density (g/cm)	6.45	8.03
Recovered Elongation	~8%	~0.8%
Young Modulus (GPa)	(M., -10°C) ~5; (A., 50°C) ~9	~200
Ult. tens. strenght (UTS, GPa)	~1.2	~0.76
Torqueability	Excellent	Poor
Biocompatibility	Excellent	Fair

MATERIALS AND METHODS

Several models of endodontic instruments are present on the world market; our investigation concerns the NiTi file produced by Maillefer Instrument SA (Ballaigues, Switzerland) known with the trade name "ProFile". These instruments have two different cone shapes: 0.04 and 0.06 mm per mm length. In this investigation we have studied the .04 ProFile numbers 15, 30 and 45 with an useful lenght of 16 mm. The number (in hundredth of millimetre) represents the diameter of the device tip base, which is lacking of the helicoidal blade. The devices are shaped as a screw, with a very sharp spiral to easily remove the dentinal tissue from the channel walls.

The Fig. 1 shows some investigated ProFiles (c) together the micro-motor employed at 300 rpm rotation (a) and the polymeric block resin used for the wear tests (b). This resin, so

called "endo training-block", simulates the dentinal tissue and contains a bent channel with the same shape and dimension of that of the dental root.
Dynamical mechanical thermal analysis (DMTA) [8] and differential scanning calorimetry (DSC) [9] were the techniques used to characterise the orthodontic NiTi alloys. In this way was possible to value the transformation temperatures (TTRs), which arising when the austenite ↔ martensite transition occurs.
On heating or cooling these alloys point out an hysteresis cycle. Conventionally A_s locates the start of the austenite formation on heating and A_f recognises the finish of this transformation. In the same manner M_s and M_f, on cooling, represent the start/finish martensite retransformation and their corresponding temperatures.
The characterisation measurements of different files have been performed using also: scanning electron microscopy (SEM), electron inducing X-ray emission (EIXE) and Auger electron specroscopy (AES). Files were tested as received and after different wear tests performed in the polymeric resin.
The mechanical measurements were conducted by an analyser (Rheometric Scientific), which was used with two heads, independently working in "bending" and "tensile" mode. All two mechanical experiments were carried out at the 3 Hz frequency. The analysis at 0.3 and 30 Hz produces the same behaviour in the dynamic modulus (E'). This evidence is characteristic of a first order transition (rather of a relaxation process) and, in this case, can be ascribed to a crystalline martensite ↔ austenite transformation.
The mechanical behaviours were studied between –30 °C and 80 °C temperature range. The "bending" mode assemblage permits to apply a sinusoidal stress to have a better control of the deformation and accuracy of the E' value of the sample. The "tensile" mode technique was used applying 4.5 N load and thermal scan rate of 2 °C/min. The "bending" mode technique was used with 4 °C/min scan rate.
The DSC (Setaram Tg-DSC 111) measures the heat absorbed or given off (endo or exothermic process respectively) by a small amount of material as it is heated and cooled through the transformation temperature. The calorimetric analysis was performed with a scan rate of 5 °C/min. The thermograms were carried out in an interval of temperature of –10 °C and +50 °C using 100 mg sample inside an aluminium crucible in a nitrogen environment.
In all the thermal cycles has been used liquid nitrogen to assure high stability and check of the temperature into the experimental chamber.
SEM was employed at 20 keV electron beam and 150 pA probe current at different magnifications. EIXE was used with an electron beam of 20 keV energy detecting the emitted characteristic X-rays with a Si(Li) detector, which permits to detect photons with energies between 1 keV and 30 keV. AES spectroscopy was performed with 3 keV electron gun. AES spectra were obtained as a function of the alloy depth, tanks to a sputtering process induced by 3 keV Ar^+ beam.
Typically, the file is used with a rotation speed of 300 turns/min (rpm) and it is employed for total rotation times below 180 s. The files wear tests are obtained using the instruments inside the channel of the endo training-block.
The times of erosion range between 5 s and about 500 s. The wear of the tools was measured by the weight loss of the training-blocks performed as a function of the time just after each 5 s step work treatment. The used balance has 10 mg sensitivity. Moreover, SEM and DMTA investigated the wear of instruments.
During the mechanical erosion of the channel in the polymeric resin, the temperature of the file was monitored "on line" with a thin thermocouple placed directly in contact with the file head.

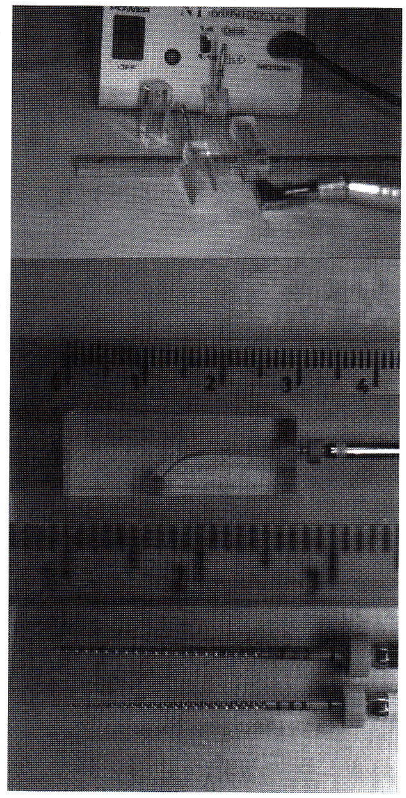
a)
b)
c)

Fig. 1: Photo of some ProFiles used in these investigations (c), file inside the endo training-block simulating the dentinal tissue channel (b) and rotary machine employed to rotate the endodontic files (a)

RESULTS

The experimental atomic composition of the ProFile alloy, measured by EIXE, is 50% Ni and 50% Ti, corresponding to the nominal values given by the producer.
Fig. 2 shows a typical spectrum of EIXE analysis (a) and AES analysis (b). The first indicate the bulk composition and the second the surface composition (the spectrum is referred to 150 nm depth). A significant amount of oxygen and carbon are present at the alloy surface, indicating that a thin carbon and TiO_2 layers cover the alloy surface, according literature [10].
The mechanical spectra of the NiTi alloy are depicted in Fig. 3a. The elastic modulus is measured as the temperature is raised and subsequently lowered. It decreases slowly starting from –20 °C up to about 10 °C (in M_f / A_s region it is approximately 8 GPa) and increases roughly from 10 °C up to about 40 °C, at higher temperature increases very slowly (its value is about 12 GPa in M_s / A_f region). The experimental TTRs behaviours are reported in Tab. II. With a scan temperature rate of 2 °C/min, starting from ~ –20 °C up to ~ 60 °C and return to –20 °C, the transformation on heating and on cooling does not overlap, i.e. a significant hysteresis has been detected as typical trend of the "shape memory" of the NiTi alloy.
The endothermic and exothermic peaks, measured by DSC and arising from heating and cooling cycle, are given in Fig. 3b. The enthalpy values of the two curves seem to be very similar (except for the sign). These results indicate that the process, connected to the aforesaid phase transition, is reversible and the TTRs values are in agreement with the previous ones given by DMTA, as reported in Tab. II. The comparison between the mechanical and calorimetric data reveals a little shift in the TTRs values, that is probably due to the different temperature ramps employed in the two cases.
Measurements of the erosion yield (removed material per unit of time) of the as received ProFiles in the polymeric resin have demonstrated that their efficiency is higher in the first 80 s use. After 120 s erosion the file surface is damaged by the wear and the erosion yield decreases of about 30-40% of

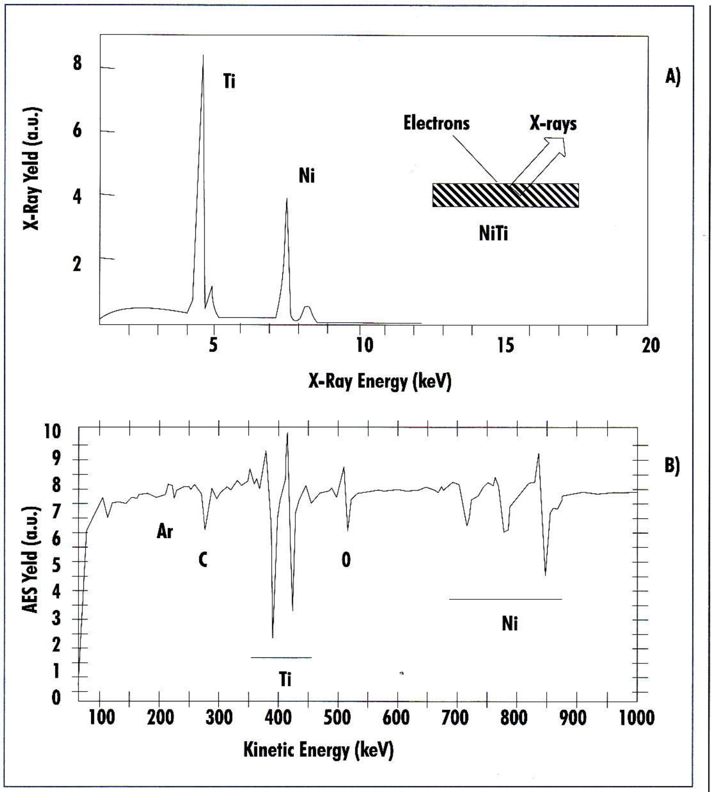

Fig. 2: Typical EIXE spectrum of NiTi alloy (a) and typical AES spectrum of the file surface analysed at 150 nm depth (45' sputtered sample) (b)

TABLE 2 - Transformation temperatures of the alloy measured by DMTA and DSC analysis

Analisys Technique	As	Af	Ms	Mf	Scan Temperature
DMTA	7	40	35	2	2°C/min
DSC	12	45	25	-5	5°C/min

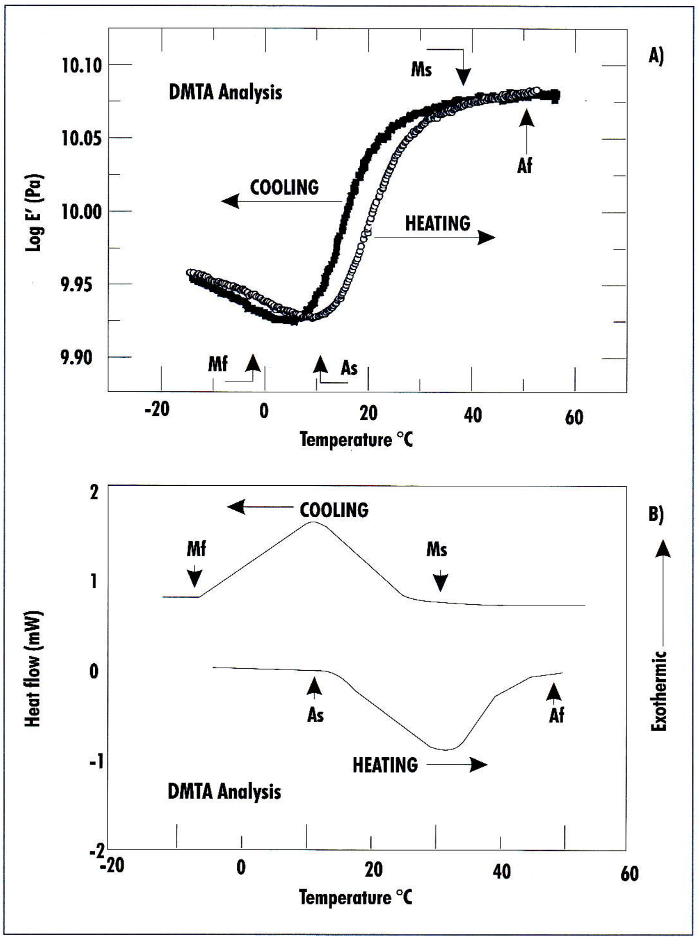

Fig. 3: Temperature behaviour of the dynamic modulus in the alloy and TTRs values and the hysteresis (a). DSC thermograms during heating and cooling run in the alloy and TTRs values and the hysteresis effects (b)

the maximum value, as reported in Fig. 4. The eroded mass shows a saturation after about 30 sec due both to the channel enlargement and to the instrument wear. Tab. III shows some results obtained using the ProFiles .04 at different conicity. The erosion yield, given in mg of removed material per 60 s erosion time, is measured always in new endo-training blocks. It is given at the beginning of the file use (new device) and after 120 s file use (old device). Obtained results demonstrate that a significant file wear occurs. More details about the erosion yield dependence with the ProFile wear have been given in our previous paper [11].

The file temperature reached during its use in the endo training-block was experimentally measured. Results indicate that the temperature increases with the time of wear is low and it reaches about 5 °C after 60 s continuum use. Starting from the body temperature, this temperature increase is not sufficient to obtain a complete transformation of the alloy in austenitic phase.

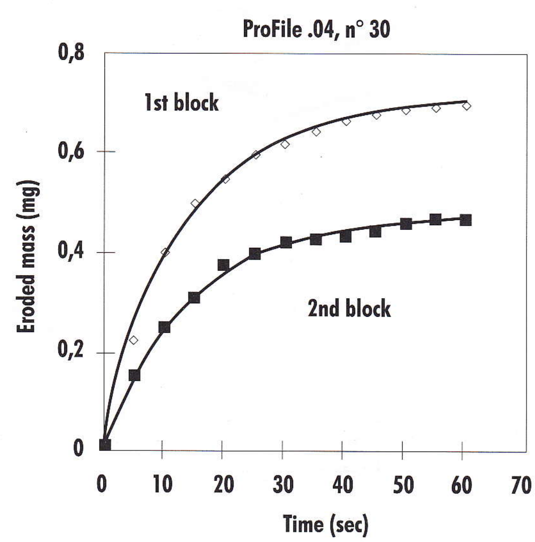

Fig. 4: Eroded resin mass by the ProFile .04 n°30 as a function of the time and wear

TABLE 3 - Erosion Yield (mg removed resin per 60 s) measured in new ProFiles so as received (new device) and after 120 s wear in resin (used devices)

ProFile Type 0.04	Erosion Yield (mg/60s) (new ProFile, as received)	Erosion Yield (mg/60s) (old ProFile, 120 s wear)
N° 15	1.5	1.2
N° 30	0.7	0.4
N° 45	0.7	0.4

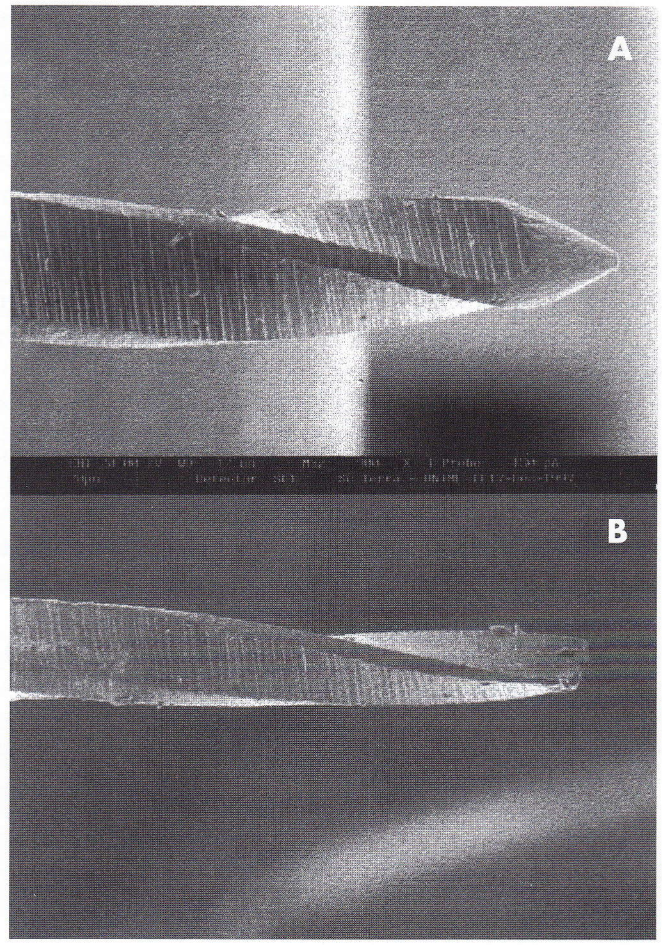

Fig. 5: SEM morphology comparison between the as received ProFile tip (a) and the file tip after 300 s wear (b)

The morphological modifications due to the wear can be observed also at the SEM, as reported in Fig. 5 with x 900 magnification. It shows the comparison between an as received ProFile with a new tip (a) and a file tip after 300 s wear indicating a full tip loss (b). At higher magnifications it is possible observe the presence of microfractures and the wear of the spiral blade responsible of the cutting loss of the device.

Another interesting result, related to the mechanical response of as received and utilised files, has been obtained by DMTA measurements and is shown in Fig.6. Analysing these spectra rises that the elastic modulus, in the martensitic zone, increases as a function of the wear time. It's well evident that the difference between martensitic and austenitic phases decreases with the wear and seems that the material become more and more rigid evolving toward to an austenitic structure. The wear produces stiffening of the alloy, with elasticity loss and increase of the rupture risk of the file.

A comparison between the mechanical behaviours of NiTi file and similar instruments, stainless steel based, was performed. It reveals that the maximum value of the NiTi elastic modulus remains about a factor five lower with respect to that of the stainless steel (~ 50 GPa). The stainless steel file shows a very high mechanical rigidity and an elastic modulus independent in all the investigated temperature range. Moreover, differently of the NiTi alloy, the dynamic modulus of the stainless steel device is independent on the device wear.

DISCUSSION AND CONCLUSIONS

At the body temperature the elastic modulus in a new ProFile is about 7 GPa and the alloy contains about 50% austenitic and 50% martensitic phases. At this temperature the material is highly elastic and flexible and suitable to be employed as guide wire.

The obtained data permit to have a better understanding of the NiTi file used in edodontic field. These instruments appear more useful than the stainless steel devices because they follow very well the dental channels also in presence of bent channels. The superelasticity of the alloy and its design permit to erode uniformly the channel with a constant cutting force and to remove efficaciously the debris. The friction of the device inside the channel damages the spiral blade of the file reducing the cut ability of the dentinal tissue.

On the base of the experience of endodontists, to reduce the risk of tip rupture inside the channel, it is advisable to observe the following recommendations:

1) to keep low the number of turns per minute of the ProFile (around 300 rpm) to maintain low the torsion stress;
2) to use adequate files, according the "crown-down" technique [12], i.e., to adapt the profile diameter to the canal diameter avoiding mismatches which induce high stresses;
3) do not use continuum rotation times higher than 20 s to maintain low the file temperature increase due to friction;
4) to change the file just found a mechanical resistance to further advancement of the instrument inside the channel to avoid that initial cracks may induce tip rupture;
5) to maintain low the file temperature working with a lubricating liquid to decrease the friction, using, for example, a jet of water (20 °C) inside the eroding channel.

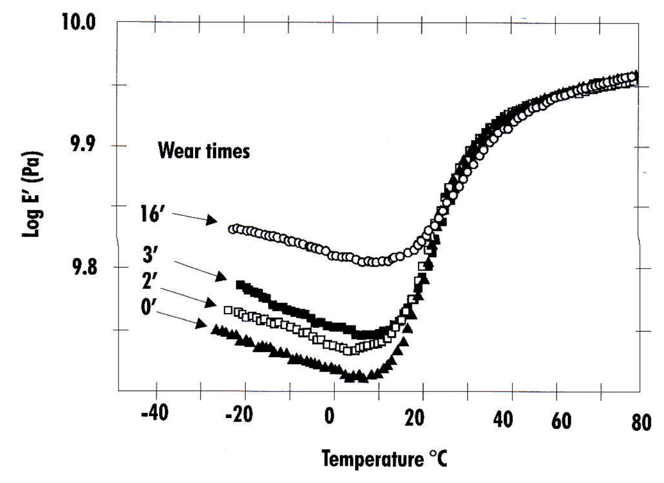

Fig. 6: Dynamic modulus of the NiTi file as a function of the temperature and of the wear time

In any case the experience demonstrates that the rupture risk of NiTi alloy files is lower than that of stainless steel files, despite these last have rotated to hand.

Recent investigations have demonstrated that the ProFiles submitted to sterilisation in autoclave, at about 140 °C in water vapour for different hours, worse their performance. Probably this effect is due to the surface oxidation increasing of the alloy, as obtained by AES analysis [13].

ACKNOWLEDGEMENTS

Authors thank Prof. E. Rapisarda and Dr. A. Bonaccorso of the Clinica Odontoiatrica I of Catania University for the useful endodontic support given to this work. Many tanks also to Dr. C. Gentile of the Physics Department of Messina University for the SEM investigations.

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