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Morphological alterations of rotary nickel-titanium protaper® instruments analyzed by SEM: effect of nitrogen ion implantation

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Abstract

Objective: To analyze, using scanning electron microscopy [SEM], the morphological changes [distortion, loss of substance, and fracture] of ProTaper SX rotary instruments submitted to nitrogen ion implantation.

Methods: Thirty ProTaper SX Instruments were divided into 3 groups of 10 instruments each. Groups A and B were subjected to ionic implantation with bands of nitrogen ions at 100 kV [group A] or 200 kV [group B] and an ion dose of 1.0×10^{17} ions/cm². Instruments in group C served as unmodified controls. Each instrument was utilized to prepare 5 canals in epoxy resin blocks with a brushing motion. Instruments were examined by SEM before use and after 60 s and 300 s of work inside canals in epoxy resin blocks.

Results: No significant morphologic alterations were observed between the groups of instruments after preparation of the simulated canals. However, whereas only 1 instrument in group A and 1 instrument in group B fractured during use, 3 instruments in group C fractured during use.

Conclusion: Ionic implantation of nitrogen ions moderately improved the performance of SX files. Instruments treated by ionic implantation at either dose yielded better results than the untreated control group, although the differences were not significant.

Key words: Endodontics; scanning electron microscopy; nickel-titanium

Alterações morfológicas em instrumentos rotatórios de níquel titânio protaper®, analisados por MEV: efeito da implantação de íons nitrogênio

Resumo

Objetivo: O presente estudo analisou, através do Microscópio Eletrônico de Varredura [MEV], as alterações morfológicas de instrumentos rotatórios ProTaper SX, submetidos à implantação de íons de nitrogênio .

Métodos: Foram utilizados trinta instrumentos ProTaper SX divididos em três grupos de 10 instrumentos cada. O grupo A foi submetido a uma dose de $1{,}0 \times 10^{17}$ íons de nitrogênio/cm² e energia de 100 KeV. O grupo B foi submetido a uma dose de $1{,}0 \times 10^{17}$ íons de nitrogênio/cm² e energia de 200 KeV. O grupo C foi composto por dez instrumentos não submetidos ao processo de implantação iônica. Cada lima instrumentou cinco blocos de canais simulados de resina acrílica, com a técnica Brushing Motion. Todos os instrumentos SX foram analisados no Microscópio Eletrônico de Varredura antes do uso, após 60 s e 300 s de uso.

Resultados: A perda de material e a distorção foram estatisticamente similares em todos os grupos. Um instrumento do grupo A, um instrumento do grupo B e três instrumentos do grupo C fraturaram durante o uso.

Conclusão: A Implantação Iônica de íons de nitrogênio melhorou moderadamente o desempenho dos instrumentos ProTaper SX. Instrumentos implantados apresentaram melhores resultados do que o grupo controle, sem diferença estatisticamente significativa.

Palavras-chave: Endodontia, Microscopia Eletrônica de Varredura, níquel-titânio

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Introduction

Stainless steel instruments do not possess the necessary flexibility to ensure safe and reliable instrumentation of root canals with complex anatomy. Although the introduction of nickel-titanium (NiTi) instruments seemed to overcome this difficulty [1], the preparation of curved root canals results in morphological alterations, including wear, disappearance of the cutting edge, microfractures, and surface defects, to the instrument surface [2-7]. New surface treatment methods, such as ion implantation and plasma nitriding, have been investigated to improve the properties of these instruments and extend their life [8-12].

Most studies of nitrogen ion implantation have investigated implantation at doses of ~10^{17} nitrogen ions/cm² [8-12]. In these studies, ion implantation produced better outcomes compared to nitrided or untreated instruments [8-12]. Nitrogen ion implantation creates a layer of titanium nitride on the surface of components by using a low-energy accelerator (~100 to 400 keV) to bombard the NiTi surface with nitrogen ions [12]. Nitrogen doses of 10^{17} ions/cm² may produce titanium nitride layers of up to 100 nm thick [12].

In this study, NiTi rotary instruments were subjected to ion implantation at different doses of nitrogen. Subsequently, the instruments were used to perform instrumentation of curve-simulated canals. Morphological alterations, including distortion, material loss, and fracture, were investigated by scanning electron microscopy (SEM) analysis after instrumentation.

Methods

Instruments and Nitrogen Ion Implantation

This study employed 30 ProTaper SX (Dentsply; Maillefer, Ballaigues, Switzerland) rotary instruments, which were equally divided into 3 groups (A, B, and C; n=10 each) and subjected or not to ionic implantation, as shown in Table 1. Instruments in groups A and B received ionic implantation a dose of 1.0×10^{17} nitrogen ions/cm² and an energy of 100 keV [group A] or 200 keV [group B] for 6 h. Nitrogen ion implantation was performed at the Laboratory of Ion Implantation of the Institute of Physics, Federal University of Rio Grande do Sul (UFRGS, Porto Alegre, Brazil). Irradiations were performed using a 500-keV High-Voltage Engineering Europe (HVEE) ion implanter. Instruments in group C were not were submitted to nitrogen ion implantation and served as unmodified controls.

For ions accelerated with 100 keV, the depth of implantation for most ions was between 50 and 20 nm. When implantation was performed at a higher energy, the concentration profile was obtained at a deeper level. For ion implantation, groups of 10 NiTi files were mounted in a base cylinder system, inclined at 30°, and coupled to a motor that kept the system rotating throughout the implantation experiment, to guarantee that the entire perimeter of the files was exposed to the ion beams. The chamber in which this group was located was fixed at the end of the line of implantation. The whole system remained in a vacuum during the experiments (at a pressure of 10^{-6} Torr).

After ion implantation, the instruments were cleaned using ultrasound, brushed with a soft bristle brush, and sterilized in an autoclave [5]. Group C, which was not treated using ion implantation, was subjected to the same cleaning and preparation processes [5]. Similar cleaning and sterilization procedures were also conducted after utilization [5].

Simulated Root Canals

The study employed 150 simulated root canals fabricated from epoxy resin (Odontofix, Ribeirão Preto, SP, Brazil). The diameter of the root canals was compatible with a #20 digital spacer [5]. Chemical and mechanical preparations were performed by a single trained operator who was blinded to the instrument group assignments [5]. Before use, new files were cleaned according to the aforementioned procedures, sterilized in an autoclave, and examined by SEM (Philips XL20) at the Center of Electron Microscopy and Microanalysis at the Lutheran University of Brazil, campus Canoas/RS, Brazil [5].

Simulated root canals were irrigated with anionic detergent solution [5]. Exploration and instrumentation of the simulated canals were performed with a 10-caliber Kerr-type manual instrument up to the actual canal length (ACL), which was determined visually. Shortly afterwards, the simulated canal was instrumented with a 15-caliber Flexofile manual instrument up to the CRT. The ProTaper S1 instrument was used until it encountered resistance, via 6 insertions of 10 seconds each in the interior of the simulator canal, up to a total time of 60 seconds [12]. Then, the SX instrument was used passively until encountering resistance, again via 6 insertions of 10 seconds each, up to a total time of 60 seconds [12]. Brushing motion was used for the SX and S1 instruments.

Table 1. Division of study groups

Groups	Number of canals	Number of instruments	Number of uses	Treatment of instruments
A	50	10	5	Ion implantation at a dose of 1.0×10^{17} nitrogen ions/cm² at 100 keV on 10 rotary instruments ProTaper SX
B	50	10	5	Ion implantation at a dose of 1.0×10^{17} nitrogen ions/cm² at 200 keV on 10 rotary instruments ProTaper SX
C	50	10	5	Not submitted to ion implantation

During the exploration and mechanical-chemical preparation steps, the simulated canal was always filled with an irrigative substance, which, at any moment, was conditioned with a 5-ml glass Lüer lock. The volume of irrigation was controlled in 2 ml of solution for each change of instrument or each insertion of the instrument in the interior of the simulated canal. The irrigation technique was performed using a Navytip needle (Ultradent, South Jordan, Utah, USA), with penetration up to 3 mm of the CRT, with the help of a cursor. The irrigation movement was of small width, parallel to the length of the axis of the simulated canal, performed with aspiration with a suction tube and new inundation of the simulated canal [5].

Instrument Performance Measures

Instruments were placed in a fixture capable of holding 6 instruments at once. Each instrument was placed in the same position, with the handle turned toward the examiner. After the first and fifth uses, the file tip and the central portion of the active tip of each instrument were analyzed by SEM at 100× and 250× magnification [5]. The micrographs were evaluated by 3 examiners experienced in SEM studies and who were previously trained to analyze the images for evidence of deformation with the assistance of a template [5].

Three criteria were used to classify the instruments: distortion of spirals [stretching, shortening, or reversal of spirals], loss of material, and fracture [5]. Scores for spiral distortion were as follows:

0 – Absence of striation, shortening, or reversion of the spire in the zone examined;
1 – Striation, shortening, or reversion involving only 1 spire in the zone examined; and
2 – Striation, shortening, or reversion involving more than 1 spire of the zone examined.

With respect to the loss of substance on the instrument's surface, the following scores were used:

0 – No loss of substance in the zone examined;
1 – Loss of substance in 1 spire in the zone examined;
2 – Loss of substance in 2 spires in the zone examined; and
3 – Loss of substance in more than 2 spires in the zone examined.

In terms of fracture, the following scores were used:

0 – Absence of fracture; and
1 – Presence of fracture. [5]

Agreement between the three examiners was evaluated using Kendall's concordance test for distortion and loss, and the Kappa test for number of fractures. Distortion and loss of material were analyzed using the non-parametric Kruskal-Wallis test. Differences with p-values $< .05$ were considered statistically significant.

Results

Examiner Agreement

Kendall's concordance test indicated good agreement between the examiners for the measures of distortion (W=0.833) and loss (W=0.727). There was excellent agreement between examiners for the measure of number of fractures (Kappa=1).

Material Loss and Distortion

Non-parametric Kruskal-Wallis tests confirmed that neither distortion (p=0.368) nor loss of material (p=.368) differed significantly among the three groups (Table 2 and 3).

Table 2. Comparison of distortion between the groups analyzed

Location of file	Use	Group	Sample											Kruskal-Wallis	
			1	2	3	4	5	6	7	8	9	10	11	Rank average	p
Half of the active part	Before use	Group A	0	0	0	0	0	0	0	0	0	0	0	16.50	
		Group B	0	0	0	0	0	0	0	0	0	0	–	16.50	1.000
		Group C	0	0	0	0	0	0	0	0	0	0	0	16.50	
	During 1st use	Group A	0	S	0	0	0	0	0	0	0	0	0	15.00	
		Group B	0	0	0	0	0	0	0	0	0	0	–	15.00	.368
		Group C	1	S	0	0	0	0	0	0	0	0	0	16.50	
	During 5th-use	Group A	0	S	0	0	0	0	0	0	0	0	0	14.50	
		Group B	0	0	F	0	0	0	0	0	0	0	–	14.50	1.000
		Group C	F	S	0	0	F	0	0	0	0	0	0	14.50	
Point	Before use	Group A	0	0	0	0	0	0	0	0	0	0	0	16.00	
		Group B	0	0	0	0	0	0	0	0	0	0	–	16.00	1.000
		Group C	0	0	0	0	0	0	0	0	0	0	0	16.00	
	During 1st use	Group A	0	S	0	0	0	0	0	0	0	0	0	16.00	
		Group B	0	0	0	0	0	0	0	0	0	0	–	16.00	1.000
		Group C	0	S	0	0	0	0	0	0	0	0	0	16.00	
	During 5th-use	Group A	0	S	0	0	0	0	0	0	0	0	0	15.50	
		Group B	0	0	F	0	0	0	0	0	0	0	–	15.50	1.000
		Group C	F	S	0	0	F	0	0	0	0	0	0	15.50	

Table 3. Comparison of loss of substance between the groups analyzed

Location of file	Use	Group	Sample											Kruskal-Wallis	
			1	2	3	4	5	6	7	8	9	10	11	Rank average	p
Half of the active part	Before use	Group A	0	0	0	0	0	0	0	0	0	0	0	16.50	
		Group B	0	0	0	0	0	0	0	0	0	0	–	16.50	1.000
		Group C	0	0	0	0	0	0	0	0	0	0	0	16.50	
	During 1st use	Group A	0	S	0	0	0	0	0	0	0	0	0	15.00	
		Group B	0	0	0	0	0	0	0	0	0	0	–	15.00	.368
		Group C	1	S	0	0	0	0	0	0	0	0	0	16.50	
	During 5th use	Group A	0	S	0	0	0	0	0	0	0	0	0	14.50	
		Group B	0	0	F	0	0	0	0	0	0	0	–	14.50	1.000
		Group C	F	S	0	0	F	0	0	0	0	0	0	14.50	
Point	Before use	Group A	0	0	0	0	0	0	0	0	0	0	0	16.00	
		Group B	0	0	0	0	0	0	0	0	0	0	–	16.00	1.000
		Group C	0	0	0	0	0	0	0	0	0	0	0	16.00	
	During 1st use	Group A	0	S	0	0	0	0	0	0	0	0	0	14.50	
		Group B	0	0	0	0	0	0	0	0	0	0	–	14.50	.387
		Group C	0	S	0	0	1	0	0	0	0	0	0	15.95	
	During 5th use	Group A	0	S	0	0	0	0	1	0	0	0	0	15.82	
		Group B	0	0	F	0	0	0	0	0	0	0	–	14.50	.441
		Group C	F	S	0	0	F	0	0	0	0	0	0	14.50	

p = Minimum significance level for the χ^2 test
S = Instruments that fractured in their 1st use were discarded and replaced by no. 11 instruments.
The "–" trace in the number 11 instrument means that, for this group, there was no fracture during the 1st use and there was no need for repositioning in group B.

In terms of absolute numbers, 2 instruments in group C exhibited loss of material compared to 1 instrument in group A and no instruments in group B. The 2 instruments not submitted to implantation were eroded after 60 s of work, whereas the ion-implanted instrument of group A lost material after 300 s of work. One instrument in group C exhibited loss of material at the tip (Figure 1a), while another was eroded at the center of the active tip (Figure 1b). One instrument in group C exhibited distortion (Figure 1b), whereas no instruments in groups A or B were distorted during use.

Fracture

One SX instrument in group A fractured after 60 s of use. One instrument in group B fractured after 300 s of use (Figure 2a). The fractured instrument from group A was discarded and replaced. Three instruments in group C fractured; one after 60 s of use and two after 300 s (Figure 2b). The instrument in group C that fractured after 60 s of use was discarded and replaced (Table 4). For all uses, the χ^2 test was used to verify the significance at the 5% level. No association was revealed between the presence of fracture and the group.

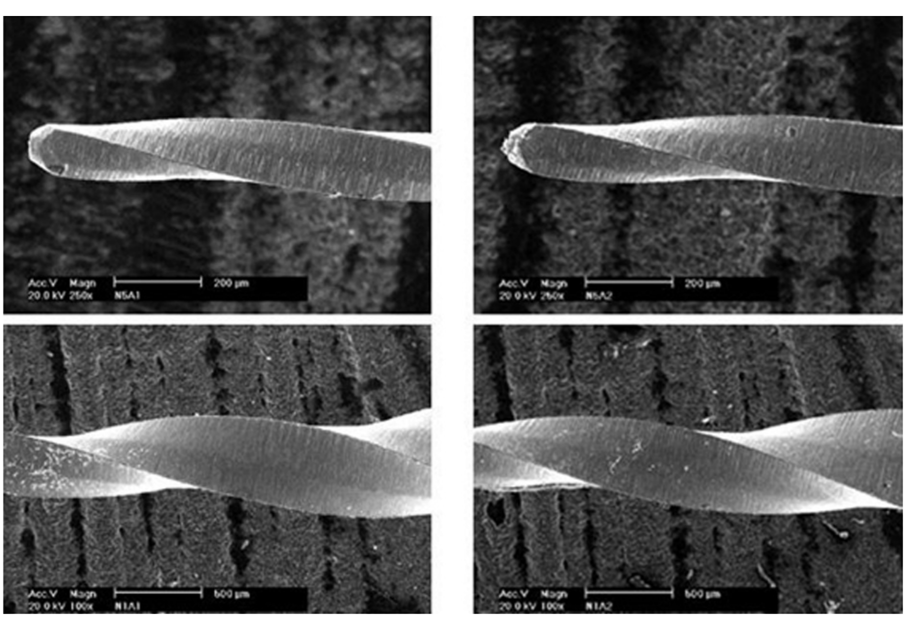

Fig 1. (a, top) SEM images of a ProTaper SX Instrument from group C, (*left*) before use and (*right*) after 60 s of use, with loss of substance on the point of the instrument (250× magnification, image width 960 μm).
(b, bottom) SEM images of an instrument in group C (*left*) before use and (*right*) after 60 s of use, displaying loss of material and distortion. Image depicts the center of the active tip (100× magnification, image width 2.4 mm).

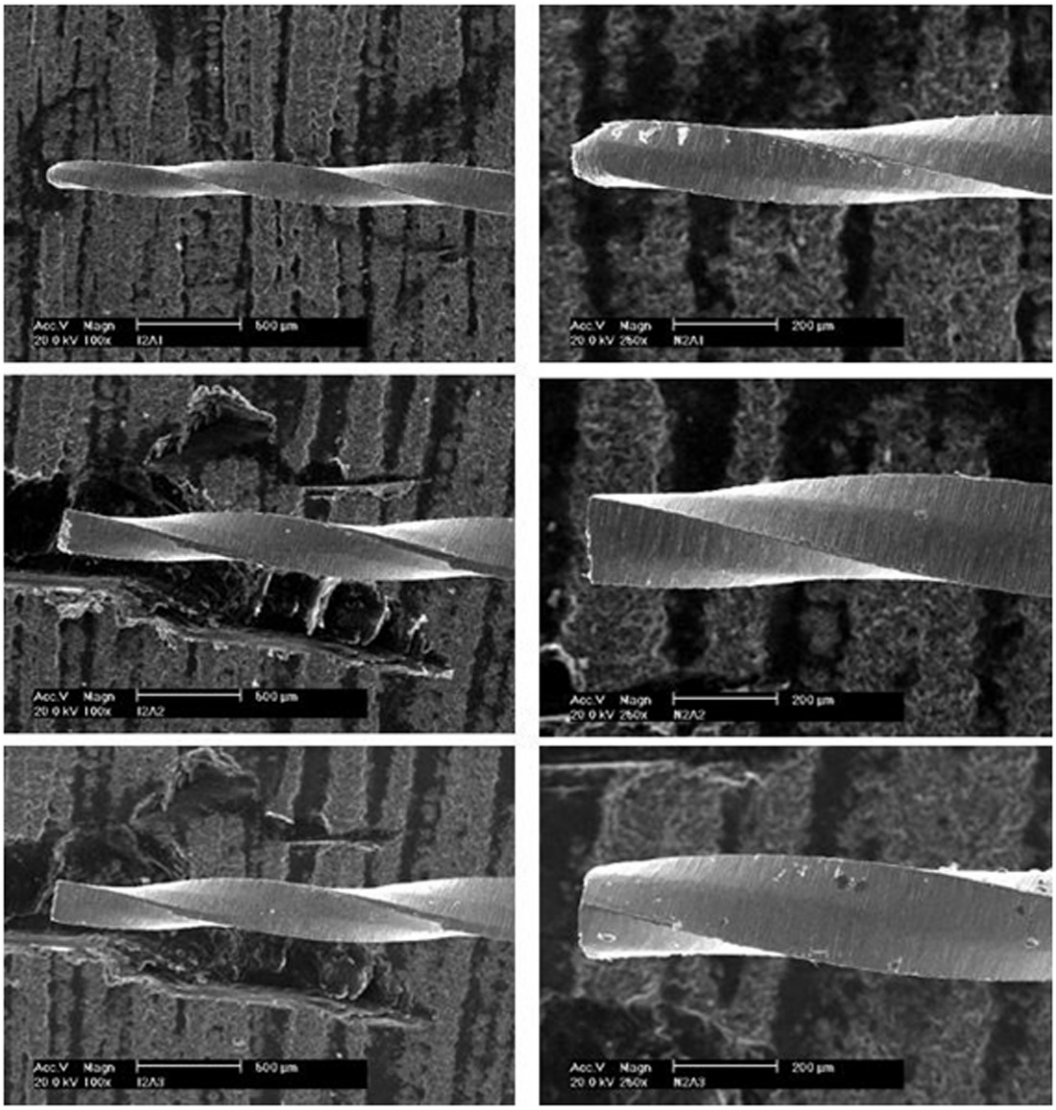

Fig. 2. **(a, Left-hand column)** SEM images of an instrument from group B, (*top*) before use, (*middle*) after 60 s of use, and (*bottom*) after 300 s of use. (100× magnification, image width 2.4 mm). **(b, Right-hand column)** SEM images of an instrument from group C, (*top*) before use, (*middle*) after 60 s of use, and (*bottom*) after 300 s of use (250× magnification, image width 960 μm).

Table 4. Comparison of fracture between the groups analyzed

Use	Group	Sample											Total fractures	p
		1	2	3	4	5	6	7	8	9	10	11		
Before use	Group 1	0	0	0	0	0	0	0	0	0	0	0	0	–
	Group 2	0	0	0	0	0	0	0	0	0	0	-	0	
	Group 3	0	0	0	0	0	0	0	0	0	0	0	0	
During 1st use	Group 1	0	1	0	0	0	0	0	0	0	0	0	1	.646
	Group 2	0	0	0	0	0	0	0	0	0	0	–	0	
	Group 3	0	1	0	0	0	0	0	0	0	0	0	1	
During 5th use	Group 1	0	S	0	0	0	0	0	0	0	0	0	0	.309
	Group 2	0	0	1	0	0	0	0	0	0	0	–	1	
	Group 3	1	S	0	0	1	0	0	0	0	0	0	2	

p = Minimum significance level for the χ^2 test
S = Instruments that fractured in their 1st use were discarded and replaced by no. 11 instruments.
The "–" trace in the number 11 instrument means that, for this group, there was no fracture during the 1st use and there was no need for repositioning in group B.

Discussion

Simulated root canals fabricated from epoxy resin were used in the present study to achieve standardization of the experimental conditions, including material hardness and curvature of the canal [5]. Given the variations in form, length, and anatomy of human root canals, many studies have opted to use extracted human teeth to produce more clinically faithful results [16]. However, despite the benefits of this approach, other studies [5-12] chose to use simulated canals, which allow for a level of standardization that cannot be obtained using extracted human teeth [17]. Moreover, epoxy-resin simulated root canals are similar to human dentin in hardness, enabling extrapolation of the experimental results to clinical practice [18]. The Knoop hardness of the resin is 220 MPa (22 kg/mm^2) [19], while the hardness of dentin is ~400 to 700 MPa (40-70 kg/mm^2) and may be reduced up to 70 MPa (7 kg/mm^2) through chelation [19].

To increase contact between the instrument and the simulated root canal and to permit better evaluation of morphological alterations and differences between groups, the rotary endodontic instruments were applied to the simulated root canals for six 10-s periods [12]. Although use of an endodontic instrumentation simulator would have provided more uniform pressure and depth across trials, in the present study, the procedure was performed by a trained specialist in endodontics to maintain the user variability present in clinical use [5]. The variable pressure that occurs during simulated root canal instrumentation may be critical to achieving the most applicable data in this work, as deformation and fracture of rotary instruments may be attributed, at least in part, to such changing pressure [5].

The fracture rate of NiTi rotary instruments has been shown to be lower after manual preflaring, resulting in a 6-fold increase in tool longevity [14]. Therefore, in this study, manual instrumentation with size 10 Kerr files and #15 Flexofile files was performed prior to use of the ProTaper instruments. The ProTaper SX instruments displayed few morphological alterations, despite having a substantial rate of tip fractures. This result may be explained by the morphology of the instruments, as the thinnest cross-sectional areas occur near the tip [20]. These findings partially agree with a prior report [21] analyzing 3 rotary instrumentation systems, in which the authors observed that ProTaper instruments displayed a statistically insignificant increase in the number of fractures, but less deformation than the other systems.

In the present study, the fracture rate of untreated instruments (group C) was 3 times higher than that of instruments in groups A and B. The greatest quantity of fractures in group C might have been caused by a failure in the operator's instrumentation technique, or may have been caused, at least in part, by the pressure applied by the operator when introducing the instrument to the simulated root canal. For example, pressure associated with inadvertent jamming of the tip into the resin walls could lead to torsional fracture [4]. Indeed, a previous study concluded that deformations in rotary instruments were related more to the mode of utilization than to the number of uses [4].

In the present study, 4 instruments suffered fractures after 60 s of use, a finding that is in agreement with Schäfer and Schlingemann's [22] observation that several NiTi instruments suffered fractures during their first use. In the present study, two instruments showed metal losses after 60 s of use and suffered fractures after 300 s of use. These results are in agreement with Svec and Powers' [23] observations of metal losses from the cutting angles of ProFile flutes and deformations after just one use, as well as Shen and colleagues' [24-25] reports indicating that the propagation of previous cracks and defects may explain instrument fracture.

Conclusions

Implantation of nitrogen ions moderately improved the performance of Sx files. Although both implantation doses yielded better results than the control group, they were not significantly different from the control group.

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