

## **Research Article**

### **Effect of heat sterilization on surface characteristics of two different Niti rotary systems – A SEM study**

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#### **ABSTRACT**

Rotary nickel-titanium instruments have attained great popularity because of their high flexibility, increased elastic limits and better resistance to bending and torsional failure compared to stainless steel instruments. Endodontic instruments come into contact with saliva, blood and infected pulp tissue and it is inevitable that they are sterilized after each use to avoid cross-infection between patients. The effect of heating and cooling cycle's occurring during heat sterilization on the physical and mechanical properties of few brands of endodontic instruments has been investigated in the past.

#### **Aims and objectives:**

The aim and objective of this study is to investigate the surface characteristics of Protaper and Race NiTi instruments at 0, 3 and 5 cycles of moist heat sterilization by using scanning electron microscopy.

#### **Materials and Methods:**

A total of 60 NiTi rotary instruments, 30 instruments belonging to two different brands, Protaper F2, 25 mm, tip size 25 NiTi rotary files (Dentsply – Tulsa dental) and Race 6 % 25 mm, tip size 25 NiTi rotary files (FKG Dentaire) were selected for this study. Of the 30 samples from each group, 10 samples were kept for observation in as received condition (control group / sub group a), 10 samples were sterilized for 3 cycles in autoclave (sub group b) and 10 samples were sterilized for 5 cycles in autoclave (sub group c). The sterilization cycles were conducted in an autoclave (Uniqueclave C-79, Confident dental equipments, India) at 121 degree C under 15 psi for 20 minutes. Samples were allowed to cool to room temperature for at least 30 minutes between cycles. All the samples were examined in a Stereoscan 440 SEM (Leica Cambridge Inc.) from 200 X to 1000 X magnification. The level of surface roughness of the specimens was rated and scored on four appearances, using a predefined scale and selected SEM pictures. The results were statistically analysed using ANOVA followed by Student Newman Keuls test.

#### **Results:**

In this study, the outer surface of all new Protaper and Race rotary instruments manifested structural defects, debris, pitting, metal strips and milling works. From the study it was found that, after 5 cycles of moist heat sterilization there is a statistically significant increase in surface roughness of both Protaper and Race NiTi instruments compared to as received samples, but no significant difference in between the two brands.

#### **Conclusion:**

The surface characteristics of the endodontic files as seen by SEM act as a very significant factor in failure and initiation of fracture. The presence of defects on the cutting edges of these instruments may be a reason for their relatively low cutting efficiency and may diminish their corrosion resistance and may make them more susceptible to fracture. These defects can act as areas of stress concentration at which crack propagation starts. Thus NiTi instruments, especially small sizes, should be used with caution. The

consequences of these surface imperfections in terms of durability and cutting efficiency in clinical use are difficult to assess and further studies are required.

**Keywords:** (endodontics, endodontic instruments, niti files, niti rotary, protaper, autoclave sterilization, scanning electron microscopy, surface characterization, surface roughness)

## Introduction

Infection control is an important issue in dentistry, mainly because of the concern over contagious diseases transmitted in clinical settings. Endodontic instruments come into contact with saliva, blood and infected pulp tissue. As the instruments are frequently reused, it is essential that they are sterilized after each use to avoid cross-infection between patients. The effect of heating and cooling cycles occurring during sterilization on the physical and mechanical properties of endodontic instruments has not been clearly stated.

Rotary nickel-titanium instruments are immensely popular because of their increased flexibility, better elastic limits and higher resistance to bending and torsional failure compared to stainless steel instruments. Rotary nickel-titanium instruments also enhance the possibility of an effective and predictable preparation of the canal. Nickel titanium instruments used for endodontic instruments contain approximately 55% nickel and 45% titanium. The alloy used is similar to the one originally used for orthodontics. These nitinol alloys exhibit two unique features that are relevant to endodontics: shape memory (SM) and superelasticity (SE). Both these features are as a result of reversible transformation of the austenite (parent phase) to the martensite (daughter phase). This phase transformation is thermoelastic, i.e. can be induced by cooling or application of stress. When the metal is heated or stress is released, the reverse transformation takes place. Machining characteristics and structural details that are imparted during the manufacturing process of endodontic instruments have an influence on their clinical performance. Several studies have been done in the past which report about changes in properties of the NiTi instruments after heat sterilization.

The purpose of this study is to assess the effect of moist heat sterilization on surface characteristics of two different brands of NiTi rotary instrument systems using scanning electron microscopy.

## AIMS AND OBJECTIVES

The aim and objective of this study is to investigate the surface characteristics of Protaper and Race NiTi instruments at 0, 3 and 5 cycles of moist heat sterilization by using scanning electron microscopy.

## MATERIALS AND METHODS

A total of 60 NiTi rotary instruments, 30 instruments belonging to two different brands were selected for this study.

Group I (n=30) Protaper F2, 25 mm, tip size 25 NiTi rotary files (Dentsply – Tulsa dental)

Group I (n=30) Race 6% 25 mm, tip size 25 NiTi rotary files (FKG Dentaire)

Of the 30 samples from each group, 10 samples were kept for observation in as received condition (control group/ sub group a), 10 samples were sterilized for 3 cycles in autoclave (sub group b) and 10 samples were sterilized for 5 cycles in autoclave (sub group c). The sterilization cycles were conducted in an autoclave (Uniqueclave C-79, Confident dental equipments, India) at 121 degree C under 15 psi for 20 minutes. Samples were allowed to cool to room temperature for at least 30 minutes between cycles.

The SEM analysis was done at Crystal growth centre, Anna University, Guindy, Chennai. 30 instruments of each group were examined in a Stereoscan 440 SEM (Leica Cambridge Inc.) from 200 X to 1000 X magnification. 10 Protaper and 10 Race instruments were observed for surface characteristics in the as received condition ( sub group a), 10 Protaper and 10 Race instruments were observed for surface characteristics after 3 cycles in autoclave ( sub group b) and 10 Protaper and 10 Race instruments were observed for surface characteristics after 5 cycles in autoclave ( sub group c). The level of surface roughness of the specimens was rated and scored on four appearances, using a predefined scale and selected SEM pictures [1].

Level 0 – absence of roughness

Level 1 – minimal roughness

Level 2 – moderate roughness

Level 3 – increased roughness

The results were statistically analysed using ANOVA followed by Student Newman Keuls test.

## RESULTS

The values for the surface roughness of the three subgroups of NiTi instruments at 0, 3 and 5 cycles of sterilization are given in table I.

**TABLE 1: Surface Roughness of Niti Instruments At 0, 3 And 5 Cycles Of Sterilization**

Samples	As received		3 cycles		5 cycles	
	Group I	Group II	Group I	Group II	Group I	Group II
1	1	1	1	1	2	3
2	1	1	2	2	2	3
3	1	1	1	1	3	2
4	1	1	1	2	2	3
5	1	1	2	1	3	2
6	1	1	1	1	3	2
7	1	1	1	2	2	3
8	1	1	1	2	2	2
9	1	1	2	1	3	2
10	1	1	1	1	2	3
<b>Mean</b>	<b>1</b>	<b>1</b>	<b>1.3</b>	<b>1.4</b>	<b>2.4</b>	<b>2.5</b>

## STATISTICAL ANALYSIS

Statistical analysis was done to compare the values within the group using ANOVA followed by Student Newman Keuls test and presented in table II and are graphically represented in figure 1.

Statistical analysis was done to compare the values within the group using ANOVA followed by Student Newman Keuls test.

**TABLE 2: Values for SEM Analysis**

S. No.	No. of cycles of Sterilization	Group I		Group II	
		Mean	SD	Mean	SD
1	0 cycles	1.00 <sup>¶</sup>	0.00	1.00 <sup>¶</sup>	0.00
2	3 cycles	1.30 <sup>¶</sup>	0.48	1.40 <sup>†</sup>	0.52
3	5 cycles	2.40 <sup>†</sup>	0.52	2.50	0.53
	p value within the brand	< 0.001*		< 0.001*	

\* - denotes significance at 1% level

¶ † - Different ¶ † between group denotes significance at 5% level

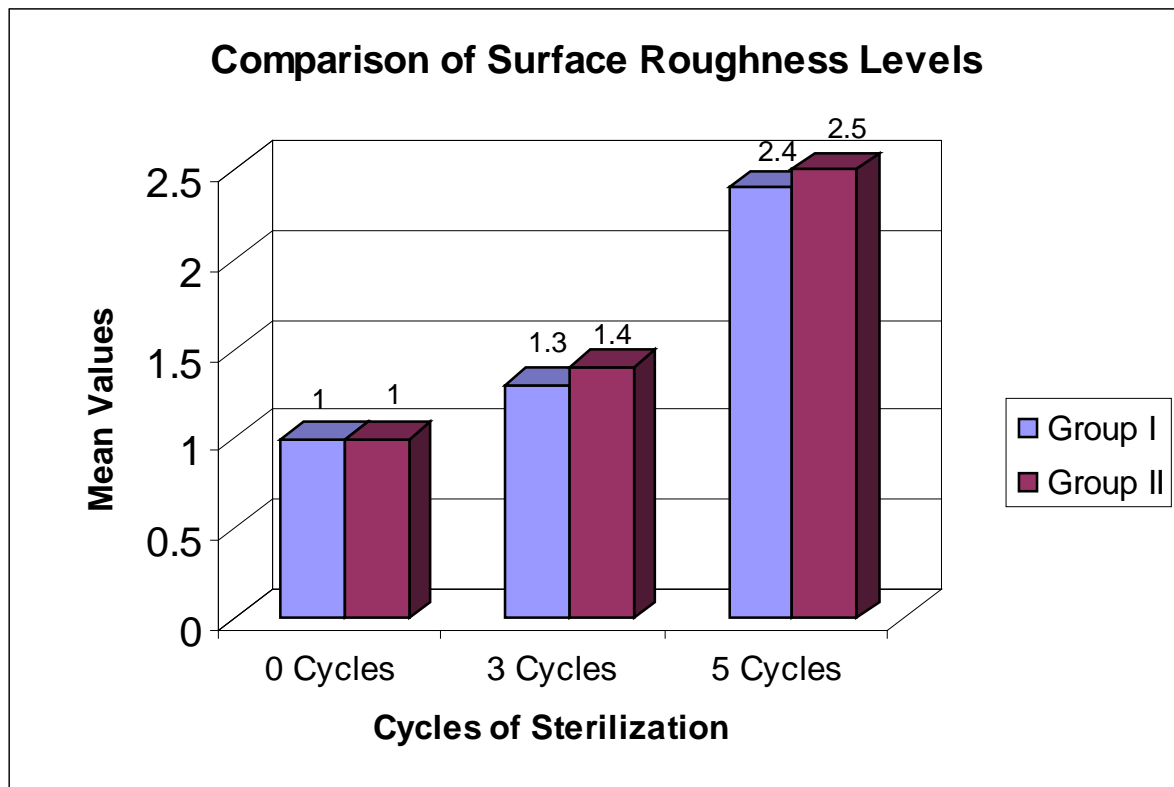


Figure 1:

## DISCUSSION

Research on developing endodontic technology is very much essential for future improvement. Better knowledge about the metallurgy of root canal files, methods of usage and evaluation of the external stresses placed on the instrument will enable the clinicians to improve the quality of root canal treatment.

Dentists are exposed to a multitude of microorganisms in the saliva and blood of patients, which may cause infectious diseases ranging from the common cold to hepatitis B and AIDS. It is inevitable for the clinicians, to use effective infection control procedures and universal precautions during the dental procedure to prevent cross contamination. Endodontic instruments should be sterilized to eliminate cross contamination between patients and to improve the success rate of the endodontic therapy. The ADA council on dental therapeutics, Council on dental practice (1988, 1996) has recommended that heat sterilization be used for all instruments that can withstand repeated exposure to required sterilization temperature.

Nitinol was developed in 1963 by William. F. Buehler at the naval ordinance laboratory in silver springs. Nitinol exhibited ideal properties for endodontic applications, such as a greater flexibility than stainless steel. In 1988, Walia et al recommended nitinol for the manufacturing of endodontic instruments, since the files made from nitinol were three times more flexible and resistant to fracture than the conventional stainless steel files [2].

The most effective and preferred methods for sterilizing endodontic instruments are the steam autoclave, dry heat oven and ethylene oxide [3]. In the present study, sterilization in steam autoclave was chosen because it is one of the most accessible methods. Dry heat oven is preferred by some clinicians for sterilization of stainless steel endodontic instruments, because these undergo corrosion in conventional steam autoclaves. But, dry heat is not as penetrating as steam autoclave sterilization, and also requires higher temperature and longer times [4,5]. Only proper steam autoclaving can reliably produce completely sterile instruments [6].

Any sterilization procedure should not significantly alter the physical properties of endodontic instruments [7]. The choice of maximum five sterilization cycles in this study was based on the literature reports, which state that Ni-Ti rotary instruments can be utilized safely to shape 10 curved root canals [8,9]. Thus, five cycles of sterilization correspond to clinical use during the average useful life of the instrument.

Scanning Electron Microscope was used to evaluate the surface changes of the instruments since it provides accurate topographic analysis of the file surface. Manufacturing of NiTi endodontic files is a complex process compared to that of stainless steel instruments. Wires containing about 55 wt% nickel and 45 wt% titanium are machined instead of twisted to produce rotary endodontic files. But, this grinding process of NiTi alloy leads to surface imperfections, especially on the cutting edges of NiTi files.

In the present study, the outer surface of all new Protaper and Race rotary instruments presented some kind of structural defects. Debris, pitting, metal strips and milling works were detected on all new instruments. The surface texture of new Race instruments was relatively smoother than that of new Protaper instruments, but at the same time race instruments had a lot of surface defects. The smoother texture was probably due to the exclusive electro-chemical treatment which the manufacturer claims of, which provides better resistance to torsion and metal fatigue.

Adherent debris was present in new files of both brands, which occurred probably from decomposition and oxidation of the lubricating oil used in manufacturing the instruments. Morphometric variations amongst the instruments of the same brand can be due to the thermo-mechanical history of each instrument during its manufacturing procedure [10,11].

These findings are in agreement with the findings of other authors that reported structural defects in new Light speed and new Profile NiTi instruments [12-14]. These findings indicate the difficulty in machining defect free NiTi instruments. The consequences of these surface imperfections on the durability and cutting efficiency of the instruments in clinical situations are difficult to

assess and have not yet been adequately evaluated.

According to Thompson, the presence of these manufacturing defects on the cutting edges of these files, is responsible for their reduced cutting efficiency and may affect their corrosion resistance too and may increase their susceptibility to fracture [10]. These defects can also serve as areas of stress concentration at which crack propagation starts. So NiTi instruments, especially small sizes, should be used with caution. In contrast, Eggert et al, claimed that the presence of these imperfections and surface irregularities are probably clinically insignificant [13]. Some surface engineering techniques have been suggested to improve surface microhardness and corrosion resistance of NiTi instruments, such as implantation of boron ion, thermal nitridation process, physical vapour deposition of titanium nitride particle ( $Ti_3N_4$ ), cryogenic treatment [15-19].

In the present study, in all the samples that were subjected to repeated sterilizations, an increased surface roughness occurred compared to unsterilized samples. A possible explanation for this might be the alterations in thickness of the passive  $TiO_2$  [titanium oxide] layer that covers NiTi surfaces.

Rapisarda et al and Thierry et al found an increase in the  $TiO_2$  oxide surface layer of NiTi alloys that were exposed to sterilization [20,21]. According to Schafer et al and Rapisarda et al, this increase in surface NiTi oxides leads to a decrease in cutting efficiency of NiTi instruments [17,20].

After scoring the specimens for surface roughness, using a predefined scale and selected SEM pictures [1], statistical analysis was done with 'ANOVA followed by student Newman Keuls test. There was no significant difference in surface roughness, between the two brands, Protaper and Race. In the case of Protaper NiTi files, analysis showed non-significant difference in surface roughness between 0 and 3 sterilization cycles and statistically significant difference between 3 and 5 sterilization cycles. Whereas in the case of Race NiTi files, there was statistically significant difference between 0 and 3 sterilization cycles as well as between 3 and 5 sterilization cycles.

## CONCLUSION

In this study, the outer surface of all new Protaper and Race rotary instruments presented structural defects, debris, pitting, metal strips and milling works. From the study it was concluded that, after 5 cycles of moist heat sterilization there is a statistically significant increase in surface roughness of both Protaper and Race NiTi instruments compared to as received samples, but no significant difference in between the two brands. The surface state of the endodontic files as viewed by SEM is an important criterion in failure and fracture initiation. The presence of defects on the cutting edges of the instruments, may be responsible for their relatively low cutting efficiency and may compromise their corrosion resistance, and may make them more prone to fracture. These defects can serve as areas of stress concentration at which crack propagation starts. Thus NiTi instruments, especially small sizes, should be used with caution. The consequences of these imperfections in terms of durability and cutting efficiency in clinical use are difficult to assess and further studies are required.

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