



СРПСКИ АРХИВ
ЗА ЦЕЛОКУПНО ЛЕКАРСТВО
SERBIAN ARCHIVES
OF MEDICINE

Address: 1 Kraljice Natalije Street, Belgrade 11000, Serbia

+381 11 4092 776, Fax: +381 11 3348 653

E-mail: office@srpskiarhiv.rs, Web address: www.srpskiarhiv.rs

Paper Accepted*

ISSN Online 2406-0895

Original Article / Оригинални рад

Milica Jovanović-Medojević^{1,†}, Martina Pelemiš², Jelena Nešković¹, Marijana Popović-Bajić¹,
Đorđe Stratimirović³, Slavoljub Živković¹

**Analysis of working surface in new manual and rotary endodontic instruments
(scanning electron microscopy)**

Анализа површине радног дела нових ручних и машинских ендодонтских
инструмената (скенинг електронска микроскопија)

¹University of Belgrade, School of Dental Medicine, Department of Restorative Dentistry and Endodontics, Belgrade, Serbia;

²Private Dental Practice AdenT, Belgrade, Serbia;

³University of Belgrade, School of Dental Medicine, Department of Basic Sciences, Belgrade, Serbia

Received: July 4, 2019

Revised: February 1, 2020

Accepted: March 6, 2020

Online First: March 10, 2020

DOI: <https://doi.org/10.2298/SARH190704018J>

***Accepted papers** are articles in press that have gone through due peer review process and have been accepted for publication by the Editorial Board of the *Serbian Archives of Medicine*. They have not yet been copy-edited and/or formatted in the publication house style, and the text may be changed before the final publication.

Although accepted papers do not yet have all the accompanying bibliographic details available, they can already be cited using the year of online publication and the DOI, as follows: the author's last name and initial of the first name, article title, journal title, online first publication month and year, and the DOI; e.g.: Petrović P, Jovanović J. The title of the article. Srp Arh Celok Lek. Online First, February 2017.

When the final article is assigned to volumes/issues of the journal, the Article in Press version will be removed and the final version will appear in the associated published volumes/issues of the journal. The date the article was made available online first will be carried over.

†**Correspondence to:**

Milica JOVANOVIĆ-MEDOJEVIĆ

School of Dental Medicine, Department of Restorative Dentistry and Endodontics, 4 Rankeova St., Belgrade 11000, Serbia

E-mail: medojevic.milica@gmail.com

Analysis of working surface in new manual and rotary endodontic instruments (scanning electron microscopy)

Анализа површине радног дела нових ручних и машинских ендодонтских инструмената (скенинг електронска микроскопија)

SUMMARY

Introduction/Objective The objective of this study is to use scanning electron microscopy to analyze working surfaces of new manual and rotary endodontic instruments and to check possible existence of manufacture dirt or defects on working surface.

Methods In this study, we used three sets of new manual instruments: K-File, KF (Dentsply Maillefer, Switzerland) and Hedstorm Files, HF (SybronEndo Co, USA) and three sets of mechanical Ni-Ti instruments - type K3 (SybronEndo Co, USA) and BioRaCe (FKG DENTAIRE Swiss Dental Products, Switzerland). Instruments were analyzed using scanning electron microscopy method at 170x magnification while semi-quantitative EDXS analysis was used to determine chemical composition of dirt particles. Fisher test ($p < 0.05$) was applied in statistical analysis.

Results showed that none of the instruments were defect-free. The most common defect type was the presence of metal strips which were noticed at the surface of all tested instruments. Debris was present on all manual and only one type of mechanical instrument, K3 (39% in apical and 33% in middle third). Fretting was noticed in all manual KF and all mechanical instruments of K3 group. Pitting was common in all manual instruments, KF (33% in apical and 39% in middle third) and HF (11% in apical and 6% in middle third). Corrosion of working surface, metal flash and disruption of cutting edge were marked only in KF group.

Conclusion Manufacture defects were noticed in all instruments and the most common type of irregularity were metal strips. Electropolished surface of BioRaCe instruments showed no debris of organic origin.

Keywords: stainless-steel manual instruments, Ni-Ti rotary instruments, defects, SEM, debris.

САЖЕТАК

Увод/Циљ Циљ овог рада је био да се применом скенинг електронске микроскопије (СЕМ) анализирају површине нових ручних и машинских ендодонтских инструмената и провери евентуално, постојање производних нечистоћа или дефеката на радном делу.

Метод У истраживању су коришћена по три сета нових ручних инструмената: *K-File*, *KF* (*Dentsply Maillefer*, Швајцарска) и *Hedstorm Files*, *HF* (*SybronEndo Co*, САД) и по три сета машинских *Ni-Ti* инструмената, типа *K3* (*SybronEndo Co*) и *BioRaCe* (*FKG DENTAIRE Swiss Dental Products*, Швајцарска). Инструменти су подвргнути СЕМ анализи са увећањем 170 \times а семиквантитативном, *EDXS* анализом, утврђиван је хемијски састав нечистоће. Статистичка анализа је урађена применом Фишеровог теста ($p < 0.05$).

Резултати су показали да не постоји ни један инструмент без дефекта. Најучесталији тип дефекта, је било присуство металних опилака, који су уочени на површини свих испитиваних инструмената. Дебри је уочен, на свим ручним и само на једном типу машинских инструмената, *K3* (39% на апикалној и 33% на средњој трећини). Жљебови су уочени на свим ручним *KF* и свим машинским инструментима *K3* групе. Присуство удубљења, забележено је код ручних инструмената, *KF* (33% апикална и 39% средња трећина) и *HF* (11% апикална и 6% средња трећина). Корозија радне површине, појава углачане површине и прекид сечивне ивице су уочени само у групи *KF*.

Закључак На свим испитиваним инструментима су уочени производни дефекти, а најучесталији тип неправилности су метални опилци. На електрополираној површини *BioRaCe* инструмената није уочено присуство органског „дебрија“.

Кључне речи: челични ручни инструменти, *Hu-Tu* ротирајући инструменти, дефекти, СЕМ, *debris*

INTRODUCTION

Clinical endodontics implies so-called “cleaning and shaping” concept while cleaning complex endodontic space from vital, necrotic or infected pulp tissue, bacteria and its products and shaping while preserving the original form of radix canal [1,2,3].

Chemomechanical root canal treatment is usually performed with manual endodontic instruments (made of stainless-steel or Ni-Ti alloy) or mechanical Ni-Ti rotary endodontic instruments with adequate and abundant irrigation of canal system. The use of rotary Ni-Ti

files in endodontic practice reduces the possibility of errors during instrumentation such as obstruction, steps, transportation and perforation of the canal wall [4].

Even though Ni-Ti rotary instruments are more efficient when compared to manual in almost every aspect (speed, simplicity and uniformity of instrumentation), their mutual defect is the possibility of deformation and fracture during instrumentation most likely due to inadequate use [5].

Fractured instrument is a serious threat to treatment, irrigation and filling of root canals and it may significantly affect the outcome of endodontic treatment [6]. Numerous studies researched the factors which can influence deformation and fracture of manual and mechanical endodontic instruments: Parashos et al. 2004 [7]; Di Fiore, 2007 [8]; Shen et al. 2009 [9]; Kosti et al. 2011 [10]; Priyanka et al. 2015 [11]; Gil et al. 2018 [12]. In 2018 Boutsikis and Lambrianidis made an outline of all these factors and grouped them into four categories: operator related factors, anatomy related factors, instrument related factors and technique/use related factors [13].

It is confirmed that endodontic instruments, due to their design and different manufacture process, may significantly impact deformation and fracture during root canal instrumentation [10]. Most of new endodontic instruments are non-sterile and can contain various metal debris, impurities and epithelial cells on their surface. The process of production of stainless steel files can lead to the presence of small metal scraps that are more or less retained at the work surfaces of the files [14].

Stainless-steel endodontic instruments are usually made by twisting of various steel profiles by longitudinal axis thus forming blades from vertical wire edges [15]. Ni-Ti rotary instruments are used in endodontic procedure due to their good mechanical properties, biocompatibility, ductility, corrosion resistance, low elastic modulus, and special characteristics such as super-elasticity and shape memory effect [16]. Production of Ni-Ti mechanical endodontic instruments is more complex when compared to the process of steel instruments manufacture. Due to memory property of Ni-Ti alloy, the majority of instruments are manufactured by engraving into milling machine rather than by twisting [15]. Despite the fact that top-notch computer technology is used to manufacture very complicated Ni-Ti instruments (CAD-CAM), surface defects including fretting, cracks, pitting and dirt are very common [15]. Irregularities at the instrument surface might increase its vulnerability to

fracture. Surface defects seem to be points of tension and can cause initiation and spread of cracks thus potentially highly contributing to possible fractures during instrument activation [13].

The aim of this study is to use Scanning Electron Microscope to analyze working surfaces of new manual and mechanical endodontic instruments and to check possible existence of manufacture dirt or defects on working surface.

METHODS

This research implied the use of three basic sets (each set consisting of 6 instruments) of new manual stainless steel instruments: K-File, KF (Dentsply Maillefer, Switzerland) and Hedstrom Files, HF (SybronEndo Co, USA) and three basic sets (each set consisting of 6 instruments) of machine endodontic instruments, type K3 (SybronEndo Co, USA) and BioRaCe (FKG DENTAIRE Swiss Dental Products, Switzerland). SEM analysis is realized in SEM-EDS laboratory of Faculty of Mining and Geology, University of Belgrade (JEOL JSM-6610LV, Japan), without any prior preparation.

Microphotographs are realized at 170x magnification but in case of noticeable changes on the instruments, for the purpose of more detailed analysis, they are realized at magnification up to 800x. Apex and middle third of instruments were analyzed from two different directions and each side of instrument was analyzed by three images.

Analysis of different irregularities and omissions during manufacture process implied the criteria proposed by Eggert et al. [17]: Score 1 – No visible defect, Score 2 – Pitting, Score 3 – Fretting, Score 4 – Micro fractures, Score 5 – Complete fracture, Score 6 - Metal flash, Score 7 – Metal strips, Score 8 – Blunt cutting edge, Score 9 - Disruption of cutting edge, Score 10 – Corrosion, Score 11 – Debris. Qualitative analysis was performed though obtained results were not quantified. Semi-quantitative EDXS analysis determined chemical composition of founded dirt.

The study was approved by the Ethics Commission of the School of Dental Medicine, University of Belgrade (36/6).

Statistical analysis of obtained results is performed by Fisher test ($p < 0.05$).

RESULTS

Obtained results are presented in Tables 1 and 2 and Figures 1–9.

Results of this study showed that all tested instruments had some kind of defect on their working surface. New manual and mechanical instruments did not show any signs of micro fractures, fractures or blunt cutting edges (Table 1).

Metal strips are the most common defect type which is noticed on surface of all tested instruments (Figure 1).

In K3 rotary group of Ni-Ti instruments, the presence of this contamination was 89% in apical and 78% in middle third. In all other groups, this defect was present in 100% of cases (Table 1, Figure 2). Fisher test statistical analysis did not show any significant differences between tested instruments and their apical or middle thirds.

Analysis of SEM microphotographs determined the contamination of working surface of tested instruments and subsequent EDXS analysis defined its chemical composition (Figure 3, Table 2). Thus, we divided instruments into two types – instruments contaminated with metal strips and instruments with debris.

Debris was present in all manual instruments: KF instruments (100% in apical and middle third) and HF instruments (56% in apical and middle third). This contamination type was marked in only one type of mechanical Ni-Ti instruments, K3 (39% in apical and 33% in middle third), while BioRaCe group did not show any presence of debris. (Table 1, Figure 4). While comparing debris in different manual instruments (KF and HF), statistically significant difference was noticed ($p = 0.0029$ in apical and $p = 0.0029$ in middle third). Also, among different mechanical Ni-Ti instruments (K3 and BioRaCe), there is a statistically significant difference in debris contamination; ($p = 0.076$ in apical and $p = 0.0191$ in middle third). Statistically significant difference was also noticed when all manual (KF and HF) instruments were compared to all mechanical rotary Ni-Ti (K3 and BioRaCe) instruments: ($p = 0.0001$ in apical and $p = 0.0001$ in middle third).

Fretting caused by manufacture was noticed in all manual instruments of KF group and all mechanical Ni-Ti instruments of K3 group, while manual HF and mechanical BioRaCe

instruments did not show any signs of fretting (Table 1, Figure 5). While comparing fretting on different manual instruments (KF and HF), statistically significant difference was noticed in apical and middle third ($p = 0.0001$ in apical and $p = 0,0001$ in middle third). In addition, among mechanical Ni-Ti instruments (K3 and BioRaCe), there is a significant statistical difference in fretting on working surface; ($p = 0,0001$ in apical and $p = 0,0001$ in middle third). Statistically significant difference in fretting was not marked when all manual instruments (KF and HF) were compared to all mechanical rotary Ni-Ti (K3 and BioRaCe) instruments (Figure 6).

Pitting was marked in apical and middle third of manual instruments, KF (33% apical and 39% middle third) and HF (11% apical and 6% middle third), though none of the groups of Ni-Ti instruments (Figure 4) showed any signs of pitting (Table 1, Figures 7 and 8). While comparing pitting in various manual instruments (KF and HF), statistically significant difference was noticed in apical third ($p = 0.0051$), which was also marked in middle third ($p = 0, 0045$). Among tested mechanical Ni-Ti instruments, there was no significant statistical difference in pitting (K3 and BioRaCe). Statistically significant difference in pitting was noticed by mutual comparison of all manual (KF and HF) and all mechanical rotary (K3 and BioRaCe) instruments: ($p = 0.0051$ in apical and $p = 0,0051$ in middle third).

Corrosion of working surface, metal flash and disruption of cutting edge were marked only in KF instruments (corrosion - 11% in apical and 17 % in middle third; metal flash - 11% in apical and 6% in middle third; disruption of cutting edge - 2% in apical). (Table1) In second group of KH manual instruments and in both groups of mechanical NI-Ti (K3 and BioRaCe) instruments, corrosion, metal flash and disruption of cutting edge were not detected (Figure 9).

DISCUSSION

Above all, success of endodontic therapy depends on proper instrumentation and biomechanical cleaning of root canal. Design of endodontic instruments, their metallurgical characteristics and surface may complicate endodontic treatment in case the instrument deforms or fractures during use. It is proved that manufacture defects might cause fracture of new instruments even during their first clinical use [18].

Results of this study showed that all analyzed instruments had at least one (or more) different defects prior to any use. Defects found on new instruments only confirm the difficulties in manufacture of specific endodontic instruments, and issues that may arise during their clinical use. New processes of instruments manufacture that aim at improving used materials, minimizing inherent defects and increasing the resistance of instruments to deformation and fracture very often remain undisclosed, usually due to manufacturers' originality and patents [19].

Fretting caused by manufacture process (due to milling), was marked in almost all tested manual steel instruments. Manufacture process of rotary Ni-Ti endodontic instruments is much more complex than the process of stainless steel instruments manufacture since they have to be mechanically treated instead of just being twisted as steel instruments. As a result, the problem is in this case even more significant. Attempts to conventionally manufacture Ni-Ti instruments by twisting the wire would probably result in their fracture therefore, shaping of these instruments is achieved by even pressure from a series of rollers applied to the nitinol wire thus defining their shape, conicity, form of blade edges i.e. design characteristics of such instruments. Difficulties during manufacture of rotary endodontic instruments and elimination of surface irregularities such as traces of milling and metal flash (especially on blades) which might compromise efficiency of instrument blade and potentially cause problems related to corrosion or fracture [18].

Presence of metal strips, which was noticed in all tested groups with the score of 100 % apart from the group K3 (78% in middle and 89% in apical third), confirms the complexity of endodontic instrument manufacture.

Pitting on working surface of instrument was noticed only in case of manual steel instruments (KF, HF), and it could be explained by specific technological process of manufacture as well as the presence of metal flash and defects on cutting edge in KF group.

Manufacturers are constantly in search of metallurgical modifications of Ni-Ti alloy thus trying to find a perfect solution that would increase the performance in terms of super-elasticity and memory properties of alloy [20]. During the process of manufacture, problems might occur due to the quality of Ni-Ti alloy since particles of oxide might stay incorporated in alloy during the manufacture process. Instrumentation and stress propagation might impact

these points and make nucleation spots for development of micro-fractures and possible instrument defects [21].

Composition of Nitinol alloy used for construction of endodontic instruments is 56% (weight) of nickel and 44% (weight) of titanium, thus achieving their equiatomic relation. Despite the fact that only one manufacturer (Dentsply, Maillefer Instruments SA, Ballaigues, Switzerland) revealed complete composition and detailed technological process of rotary instruments manufacture, it is assumed to be the only relation between the elements that gives alloy its super-elastic characteristic. Variation of Ni-Ti alloy composition enables manufacture of instruments with different properties: either super-elastic alloy or better memory property. Increasing the percentage of nickel or replacing the elements in traces (e.g. cobalt), results in decreased temperature of transformation. Increase in annealing temperature increases the transformation temperature as well [18].

Results of this study showed the presence of debris in all instruments apart from BioRaCe group of instruments. Such result is explained by the existence of electrochemically treated working surface of BioRaCe instrument, thus achieving better cutting edge efficacy and resistance to wear and tear. Electrochemical polishing decreases irregularities at the surface of instruments, thus decreasing the accumulation of organic debris [22, 23].

In order to decrease surface defects and improve resistance of endodontic instruments, various methods are applied: alloy implantation process by argon, boron or nitrogen ions, thermal nitridation (coating of instrument with layer of titanium nitride), plasma immersion, deep dry cryogenic treatment and electro polishing [20, 22–25]. Many researchers were focused on increasing efficiency and flexibility and decreasing resistance to cyclic fatigue. As a result, they suggest additional thermo mechanical treatment of raw Ni-Ti alloy or even finished instruments [25, 26, 27]. Process of vapor accumulation enables treatment of Ni-Ti instruments with layer of titanium nitride, thus achieving better cutting-edge efficiency and resistance to corrosion [27]. Memory shape alloys based on the equiatomic Ni-Ti composition, have a great importance in the development of modern endodontic rotary instruments [16]. It is necessary and very important to be familiar with metallurgical characteristics of Ni-Ti alloy in order to use their clinical potential in the best possible way and minimize frustration and fear that many dentists have that such instruments could fracture during root canal treatment [28, 29].

CONCLUSION

Based on the results of this research, it is concluded that all tested instruments showed manufacture defects (one or more), and that the most common defect type was the presence of metal strips on working surface of instrument. Due to the presence of debris on working surface of instrument, it is necessary to sterilize the instruments before initial use. Electropolished surface of BioRaCe instruments showed no presence of organic debris. These facts could be a warning sign to all practitioners – before initial use, working surface of instruments should be well analyzed in order to avoid possible complications during endodontic treatment.

ACKNOWLEDGEMENT

The research from this article is part of the doctoral dissertation titled *Analysis of deformation and fragment breakage of rotary nickel-titanium instruments after preparation of different root canal systems*, by Mićica Jovanović-Medojević.

Conflict of interest: None declared.

REFERENCES

1. Schilder H. Cleaning and shaping the root canal. *Dent Clin North Am.* 1974;18:269–96. [PMID: 4522570]
2. Nešković I, Stojanac I, Vujkov S, Laban Terzija J, Maletin A. Deformation of Nickel-Titanium (NiTi) rotary instruments in cyclic loading – in vitro study. *Stomatol Glas Srb.* 2018;65(2):61-70. [DOI: 10.2478/sdj-2018-0006]
3. Živković S, Jovanović-Medojević M, Nešković J, Popović Bajić M, Živković Sandić M. Efficiency of XP Endo Shaper (XPS) and Irrigation Protocol on the Quality of Cleaning the Apical Third of Root Canal: SEM Study. *Balk Dent J Med.* 2019;23(1):31-5. [DOI: 10.2478/bjdm-2019-0006]
4. Brkanić T, Stojšin I, Vukoje K, Živković S. SEM analiza kvaliteta čišćenja zidova kanala korena zuba nakon primene niki-titanijumskih instrumenata. *Srp Arh Celok Lek.* 2010;138(9-10):551-6. DOI:10.2298/SARH1010551B PMID: 21180083
5. Bakhai D, Hedge V. Comparison of surface characteristics of apical third of rotary niti files manufactured from different phases of NiTi before and after use: An SEM analysis. *DJAS.* 2016;4(1):18-22. DOI: 10.1055/s-0038-1672040
6. McGuigan MB, Louca C, Duncan HF. Clinical decision-making after endodontic instrument fracture. *Br Dent J.* 2013;214(8):395-400. DOI: 10.1038/sj.bdj.2013.379 PMID: 23619858
7. Parashos P, Gordon I, Messer H.H. Factors Influencing Defects of Rotary Ni-Ti Endodontic Instruments After Clinical Use. *J Endod.* 2004;30(10):722-5. PMID: 15448468 DOI: 10.1097/01.DON.0000129963.42882.C9
8. Di Fiore P.M. A Dozen ways to prevent Ni-Ti rotary instruments fracture. *JADA* 2007;138(2):196-201 PMID: 17272374 DOI:10.14219/jada.archive.2007.0136
9. Shen Y, Haapasalo M, Shun-pan Cheung G, Peng B. Defects in Nickel-Titanium Instruments after Clinical Use. Part 1: Relationship between Observed Imperfections and Factors Leading to Such Defects in a Cohort Study. *J Endod.* 2009;35:129-132 PMID: 19084142 DOI:10.1016/j.joen.2008.10.014
10. Kosti E, Zinelis S, Molyvdas I, Lambrianidis T. Effect of root canal curvature on the failure incidence of ProFile rotary Ni-Ti endodontic instruments. *Int Endod J.* 2011;44:917-25 PMID: 21658071 DOI:10.1111/j.1365-2591.2011.01900.x
11. Priyanka J, Nayan S, Keswani P.S. Clinical Update on Nickel- Titanium Rotary Instruments. *World J Dent.* 2015;6:97-103 DOI: 10.5005/jp-journals-10015-1322
12. Gil J, Rupérez E, Velasco E, Aparicio C, Manero JM. Mechanism of fracture of NiTi superelastic endodontic rotary instruments. *J Mater Sci Mater Med.* 2018;29(8):131. PMID: 30088100 DOI: 10.1007/s10856-018-6140-7
13. Boutsoukis C., Lambrianidis T. Factors Affecting Intracanal Instrument Fracture. In: Lambrianidis T. (eds). *Management of Fractured Endodontic Instruments.* Springer, Cham; 2018. p. 31-60. DOI: 10.1007/978-3-319-60651-4_2
14. Popović J, Gašić J, Radičević G. The Investigation of Ultrasound Efficacy in Cleaning the Surface of New Endodontic Instruments. *Srp Arh Celok Lek.* 2009;137(7-8):357-62. PMID: 19764588 DOI: 10.2298/SARH0908357P
15. Thompson SA. An overview of nickel-titanium alloys used in dentistry. *Int Endod J.* 2000;33(4):297-310. PMID: 11307203 DOI: 10.1046/j.1365-2591.2000.00339.x
16. Jokanović J, Čolović B, Živković-Sandić M. Osnovne karakteristike i primena legura sa efektom pamćenja oblika u ortodonciji i endodonciji. *Stomatol Glas Srb.* 2019;66(1):29-35. DOI:10.2478/sdj-2019-0004
17. Eggert C, Peters O, Barbakow F. Wear of Nickel-Titanium Lightspeed Instruments Evaluated by Scanning Electron Microscopy. *J Endod.* 1999;25(7):494-7. PMID: 10687515 DOI: 10.1016/S0099-2399(99)80289-1
18. Patel D, Bashetty K, Sirekha A, Archana S, Savitha B, Vijay R. Scanning electron microscopic evaluation of the influence of manual and mechanical glide path on the surface of nickel-titanium rotary instruments in moderately curved root canals: An in-vivo study. *J Conserv Dent.* 2016;19(6):549-554. PMID: 27994317 DOI: 10.4103/0972-0707.194035

19. Pongione G, Giansiracusa A, Lisotti F, Milana V, Testarelli L. Mechanical properties of endodontic instruments with different nickel-titanium alloys: a stiffness test. *ENDO*. 2012;6(1):41-4.
20. Gavini G, Santos MD, Caldeira CL, Machado MEL, Freire LG, Iglecias EF, Peters OA, Candeiro GTM. Nickel-titanium instruments in endodontics: a concise review of the state of the art. *Braz Oral Res*. 2018;32(suppl 1):e67. PMID: 30365608 DOI: 10.1590/1807-3107bor-2018.vol32.0067
21. Alapati SB, Brantley WA, Svec TA, Powers JM, Nusstein JM, Daehn GS. SEM observations of nickel-titanium rotary endodontic instruments that fractured during clinical use. *J Endod*. 2005;31(1):40-3. PMID: 15614004 DOI: 10.1097/01.DON.0000132301.87637.4A
22. Pedulla E, Savio FL, Boninelli S, Plotino G, Grande NM, La Rosa G, Rapisarda E. Torsional and Cyclic Fatigue Resistance of a New Nickel-Titanium Instrument Manufactured by Electrical Discharge Machining. *J Endod*. 2016;42(1):156-9. PMID: 26586518 DOI: 10.1016/j.joen.2015.10.004
23. Inan U, Gurel M. Evaluation of surface characteristics of rotary nickel-titanium instruments produced by different manufacturing methods. *Niger J Clin Pract*. 2017;20(2):143-146. PMID: 28091427 DOI: 10.4103/1119-3077.164342.
24. Zupanc J, Vahdat-Pajouh N, Schäfer E. New thermomechanically treated NiTi alloys - a review. *Int Endod J*. 2018;51(10):1088-1103. PMID: 29574784 DOI: 10.1111/iej.12924.
25. Kaval ME, Capar ID, Ertas H. Evaluation of the Cyclic Fatigue and Torsional Resistance of Novel Nickel-Titanium Rotary Files with Various Alloy Properties. *J Endod*. 2016;42(12):1840-1843. PMID: 27776878 DOI: 10.1016/j.joen.2016.07.015
26. Capar ID, Kaval ME, Ertas H, Sen BH. Comparison of the cyclic fatigue resistance of 5 different rotary pathfinding instruments made of conventional nickel-titanium wire, M-wire, and controlled memory wire. *J Endod*. 2015;41(4):535-8. PMID: 25510316 DOI: 10.1016/j.joen.2014.11.008
27. Plotino G, Grande NM, Mercadé Bellido M, Testarelli L, Gambarini G. Influence of Temperature on Cyclic Fatigue Resistance of ProTaper Gold and ProTaper Universal Rotary Files. *J Endod*. 2017;43(2):200-202. PMID: 27989581 DOI: 10.1016/j.joen.2016.10.014
28. Shen Y, Haapasalo M, Shun-pan Cheung G, Peng B. Defects in Nickel-Titanium Instruments after Clinical Use. Part 2: Fractographic Analysis of Fractured Surface in a Cohort Study. *J Endod*. 2009;35(1):133-6. PMID: 19084143 DOI: 10.1016/j.joen.2008.10.013.
29. Tewari RK, Kapoor B, Kumar A, Mishra SK, Mukhtar-Un-Nisar-Andrabi S. Fracture of rotary nickel titanium instruments. *J Oral Res Rev* 2017;9:37-44. DOI: 10.4103/jorr.jorr_40_16

Table 1. Presence of defects and dirt on working surface of tested instruments

Defects	KF		HF		K3		BioRaCe	
	Apical third	Middle third	Apical third	Middle third	Apical third	Middle third	Apical third	Middle third
Pitting	6	7	2	1	/	/	/	/
Fretting	18	18	/	/	18	18	/	/
Metal-flash	2	1	/	/	/	/	/	/
Metal-strips	18	18	18	18	16	14	18	18
Disruption of cutting edge	1	/	/	/	/	/	/	/
Corrosion	2	3	/	/	1	1	/	/
Debris	18	18	10	10	7	6	/	/

HF – Hedstorm files; KF – K-file

Table 2. Chemical composition of impurities (spectrums 1 and 2) and clean instrument K-3 (spectrum 3); chemical analyses are given in wt.% and are normalized to 100 wt.%

Spec.	C	N	O	Na	Mg	Al	Si	S	Cl	K	Ca	Ti	Fe	Ni	Total
Spec.1	47.7	0	37.4	2.3	1.0	0.2	2.5	0.9	3.6	1.6	1.1	0.6	0	1.1	100.0
Spec.2	49.9	0	34.1	0.7	0.6	0.4	1.8	0.3	0.5	0.4	7.6	2.0	0	1.7	100.0
Spec.3	0	0	0	0	0	0	0	0	0	0	0	44.3	0	55.7	100.0

Paper accepted

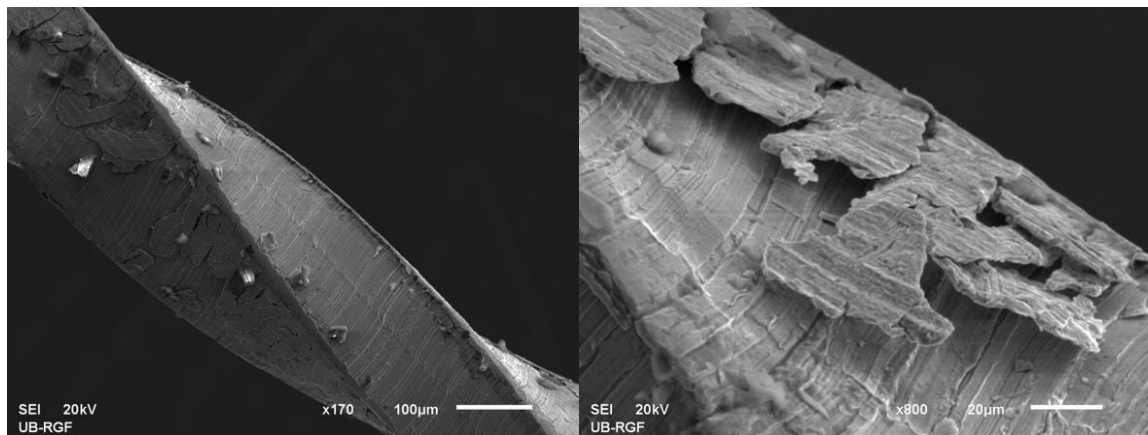


Figure 1. (a) Scanning electron microscopy analysis of K-File instrument working surface (middle third) with metal strips and fretting (magnification 170×); (b) detail from picture (a), metal strips on working surface of K-File instrument (magnification 800×)

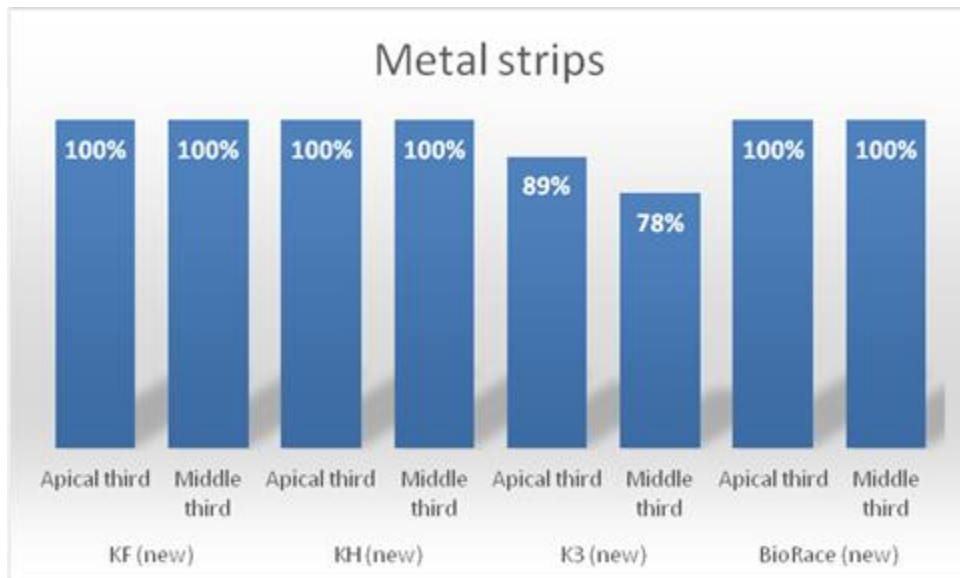


Figure 2. The presence of metal strips on working surface of tested instruments

Paper accepted

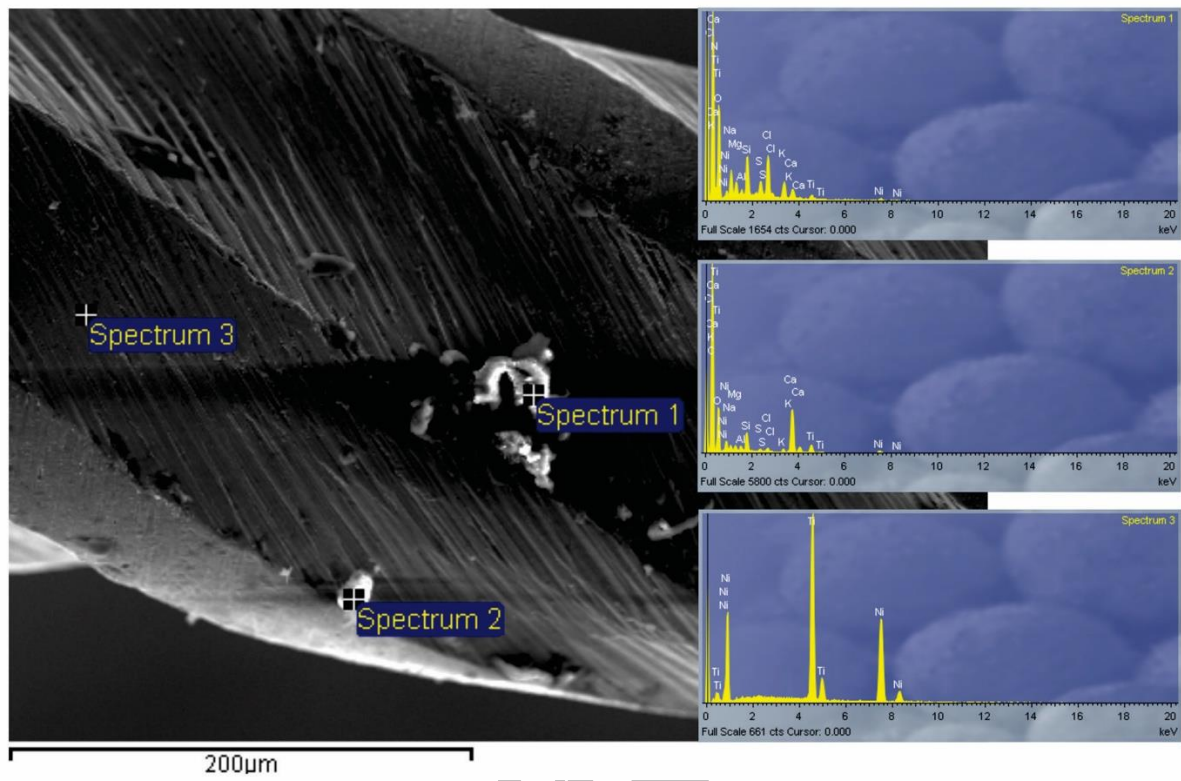


Figure 3. Energy-dispersive X-ray spectroscopy analysis

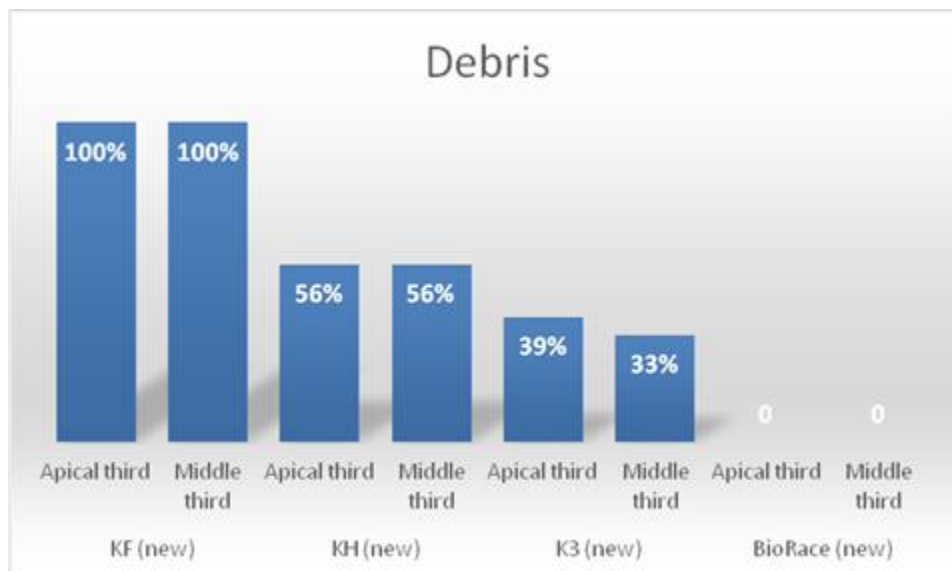


Figure 4. The presence of debris on working surface of tested instruments

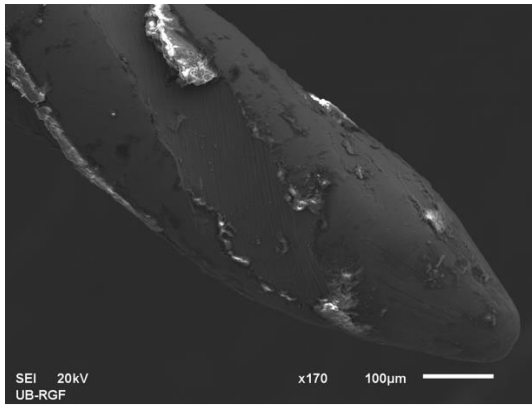


Figure 5. Scanning electron microscopy analysis of working surface of K3 instruments (apical third) with metal strips, fretting and debris (magnification 170 \times)

Paper accepted

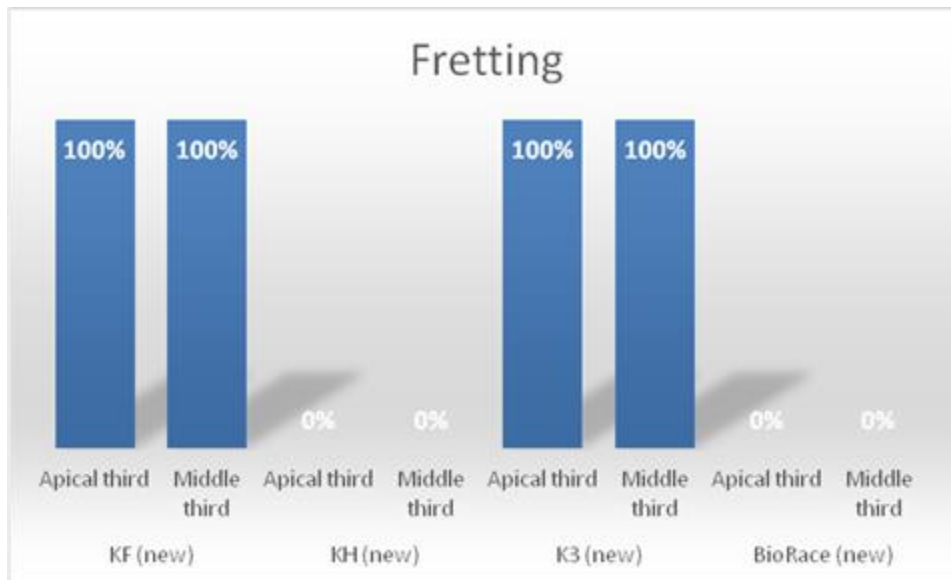


Figure 6. The presence of fretting on working surface of tested instruments

Paper accepted

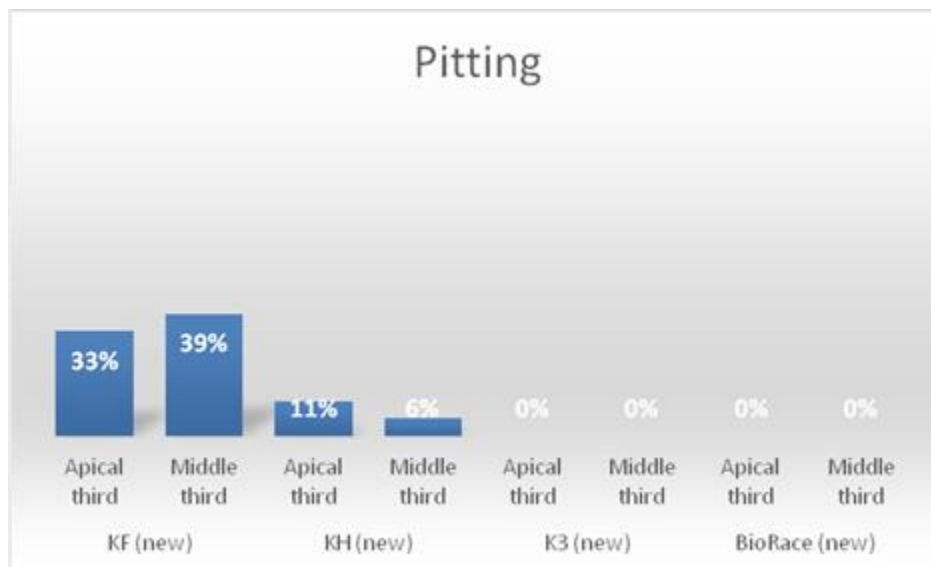


Figure 7. The presence of pitting on working surface of tested instruments

Paper accepted

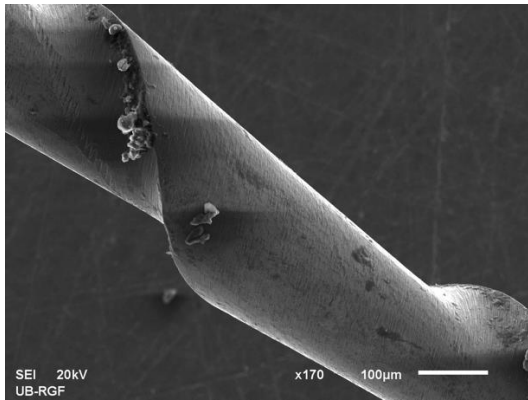


Figure 8. Scanning electron microscopy analysis of working surface of Hedstrom file instrument (middle third) with pitting and debris (magnification 170×)

Paper accepted

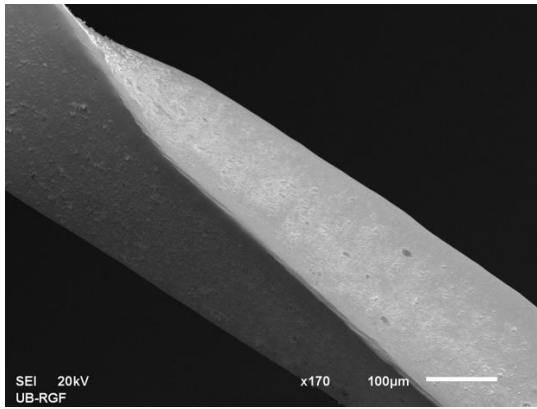


Figure 9. Scanning electron microscopy analysis of working surface of BioRaCe instrument (middle third) with small amount of metal strips on electro polished surface (magnification 170×)

Paper accepted