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Evaluation of surface characteristics of new rotary nickel-titanium instruments – SEM-EDS analysis

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SUMMARY

Introduction Modern endodontic procedure implies the use of rotary Ni-Ti instruments during chemomechanical treatment of root canals. The aim of this study is to analyze the surfaces of new (unused) rotary endodontic instruments using the SEM-EDS method and determine how frequently manufacture defects or impurities appear on their working surfaces.

Material and method Five new different sets of rotary endodontic Ni-Ti instruments were included in this study: K3, Mtwo, ProTaper Universal, HyFlex and BioRaCe. The working part of endodontic instrument was analyzed using SEM-EDS method (magnifications ×150 to ×2000), which determined the morphological characteristics of the instrument surface and chemical composition of the found impurities. Statistical analysis was performed using the Fisher's test ($p < 0.05$).

Results The results of SEM-EDS analysis showed that there is no new instrument without defects on its surface. The most common defects were observed in K3 (27.43%) and ProTaper Universal group (27.21%) and the least were in BioRaCe instruments (7.67%). The most common type of defect in tested instruments was fretting. In addition, the presence of debris and metal strips was found on all instruments, while corrosion of the working part was observed only in K3, ProTaper Universal and Mtwo systems in a small percentage.

Conclusion Based on the results of this research, it can be concluded that manufacturing defects were noticed in all examined instruments. The most common defect is pitting. Impurities such as debris and metal strips have also been registered. No organic debris was observed on electropolished surface of BioRaCe instruments, but a small percentage of other types of defects were registered.

Keywords: Ni-Ti instruments; manufacturing defects; SEM-EDS

INTRODUCTION

The application of rotary nickel-titanium (Ni-Ti) instruments with shape memory properties, biocompatibility and corrosion resistance, has introduced a new era into the endodontic procedure. Rotary Ni-Ti instruments have enabled faster and more efficient preparation i.e. reduced the possibility of procedural errors during cleaning and shaping of root canals of different morphology [1]. Numerous innovations in instrument design, in recent years related to the surface and heat treatment of Ni-Ti alloys, have affected the efficiency and required safety during endodontic treatment [2–4].

In the production of endodontic instruments, Ni-Ti alloys are used in the ratio 56: 44 = Ti: Ni, which achieves their equiatomic relationship. Although only one manufacturer (Dentsply, Maillefer Instruments SA, Ballaigues, Switzerland) published the absolute composition and detailed technological manufacturing process, it is assumed that this is the best ratio that gives the alloy super-elastic properties [5].

The production of Ni-Ti rotary endodontic instruments is much more complicated compared to the process of making steel instruments by cold twisting of pre-profiled wire cones [2]. Ni-Ti instruments are created by specific grinding, i.e. by carving a certain profile into the central stem of the Ni-Ti wire [5]. Newer production techniques include a combination of heat treatment of alloy and simultaneous twisting, for greater flexibility and better resistance to torsion and cyclic fatigue [3]. Although modern computer technology is used in the process of making a complicated design of Ni-Ti instruments, surface defects often occur in the form of fretting, pitting, cracks and impurities that can increase vulnerability to fracture [6]. It has been observed that surface defects act as points of stress concentration, leading to the initiation and spread of cracks i.e. frequent fractures during instrument activation [7].

Various metal residues and impurities of organic and inorganic origin can be found on the surface of new endodontic instruments. During instrumentation, these metal shavings can be incorporated into dentinal wall or pushed into periapical tissue and cause an allergic reaction [8]. The use of instruments with organic impurities also carries the risk of potential cross-infection [9].

The aim of this study is to analyze the surfaces of new rotary endodontic instruments using Scanning Electron Microscopy with Energy-Dispersive Spectrometry

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Table 1. Characteristics of tested sets of Ni-Ti rotary instruments
Tabela 1. Osnovne karakteristike ispitivanih setova Ni-Ti rotirajućih instrumenata

Instrument Manufacturer Instrument Proizvođač	Activation Aktivacija	Cross-section Special Features Poprečni presek, specifičnosti dizajna	Diameter Dijametar	Taper Koničnost	Manufacturing Proces proizvodnje
K3, SybronEndo	Rotary centric Puna rotacija	Triple-fluted, Positive rake angle with asymmetric radial lands Trostruka sečiva sa pozitivnim uglom i asimetričnim radijalnim površinama	25	0.12-0.02	Micromilling, conventional Ni-Ti alloy Mikroglodanje, konvencionalna Ni-Ti legura
Mtwo, VDW	Rotary centric Puna rotacija	S-shaped with two active cutting edges S-oblika sa dva aktivna sečivna ugla	10-35	0.04-0.05, 0.06	Micromilling, Conventional Ni-Ti alloy Mikroglodanje, konvencionalna Ni-Ti legura
ProTaperUniversal Dentsplay-Sirona	Rotary centric Puna rotacija	Convex triangular, Variable and progressive tapers along the instrument Konveksni trougao, varijabilna progresivna koničnost duž instrumenta	17-30	Regressive Tapers Regresivna koničnost	Micromilling, Conventional Ni-Ti alloy Mikroglodanje, konvencionalna Ni-Ti legura
HyFlex CM Coltene	Rotary centric Puna rotacija	Double fluted Hedstroöm design with positive rake angle Dvostruki Hedstrom dizajn sa pozitivnim upadnim uglom	20-40	0.04, 0.06, 0.08	Micromilling, Post- manufacture heat treatment: CM-wire Mikroglodanje CM-žica
BioRaCe, FKG	Rotary centric puna rotacija	Triangular with alternating cutting edges along the instrument Trougaoni sa alteracijama sečivnih ivica duž instrumenta	15-40	0.04, 0.05, 0.06, 0.08	Micromilling, Electropolishing Conventional Ni-Ti alloy Mikroglodanje, konvencionalna Ni-Ti legura, elekropolirana površina

(SEM-EDS) to determine how frequently manufacture defects or impurities appear on their working surfaces.

MATERIAL AND METHOD

The study used three basic sets (each set of six instruments) of five different systems of rotary endodontic Ni-Ti instruments: K3 (SybronEndo Co, USA), Mtwo (VDW, Munich, Germany), ProTaper Universal (Dentsply Maillefer, Switzerland), HyFlex (Coltene Whaledent group, Switzerland) and BioRaCe (FKG DENTAIRE Swiss Dental Products, Switzerland) (Table 1).

Scanning Electron Microscopy with Energy-Dispersive Spectrometry (SEM-EDS) was performed in the Laboratory for SEM, Faculty of Mining and Geology, University of Belgrade, using the type of SEM - JEOL JSM-6610LV, Japan. The instruments were analyzed without any preparation (directly from the factory packaging). Images were made using Secondary Electron Detector (SE images – second electron) at magnifications ranging from 150x to 2000x. Chemical analysis was performed on unpolished samples using the EDS detector (type X-Max Large Area Analytical Silicon Drifted spectrometer, Oxford Instruments) using internal standards. Obtained chemical composition is presented as the content of chemical elements in weight percent (wt%), normalized to 100%. Detection limit for most elements was about 0.1 wt%. This type of chemical analysis is considered semi-quantitative, because it was performed on unpolished surfaces.

A total of 540 recordings of apical and middle thirds of instruments were made from two different directions. Three SE images were taken for each surface of the instrument. Two researchers reviewed the images and their results were reconciled by Cohen Kappa analysis.

A qualitative analysis of various irregularities and errors present on the working surface of Ni-Ti instruments was applied in accordance with recommendations of Kristina Egert et al. [10]. The instruments registered the presence of: pitting, fretting, microfractures, complete fractures, metal flash, metal strips, blunt cutting edge, disruption of cutting edge, corrosion and presence of debris.

Statistical analysis of obtained results was performed using the Fisher's test ($p < 0.05$).

RESULTS

The results of SEM-EDS analysis of the new Ni-Ti sets are presented in Tables 2-4 and Figures 1-9.

The analysis of SE images showed the existence of surface contamination on the working part of tested instruments, and the subsequent EDS analysis determined its chemical composition. This way, a division was made into instruments contaminated with debris and instruments contaminated with metal strips. Examples of SEM-EDS analysis with the appearance of performed spectrum are presented in Figures 1 and 2.

SEM-EDS analysis in point 1 (spectrum 1, Figure 1, Table 2) shows that dominant element in the examined sample was carbon (88.1 wt%) with low oxygen content (1.5 wt%), and impurity on this ProTaper Universal instrument is characterized as a debris of organic origin. Nickel and titanium contents (spectrum 1) reflect the composition of instrument.

Based on SEM-EDS nalysis, the impurities on K3 instrument are characterized as a combination of debris of organic origin and contamination with metal strips. The quantity of nickel and titanium in analysis 1 (spectrum 1, Figure 2) and slightly more quantity of these two elements in analysis 2 (spectrum 2, Figure 2) represents the distribution of these elements in the structure of instruments, just like in the previous case.

The results of SEM analysis indicate the most frequent occurrence of defects and impurities in systems

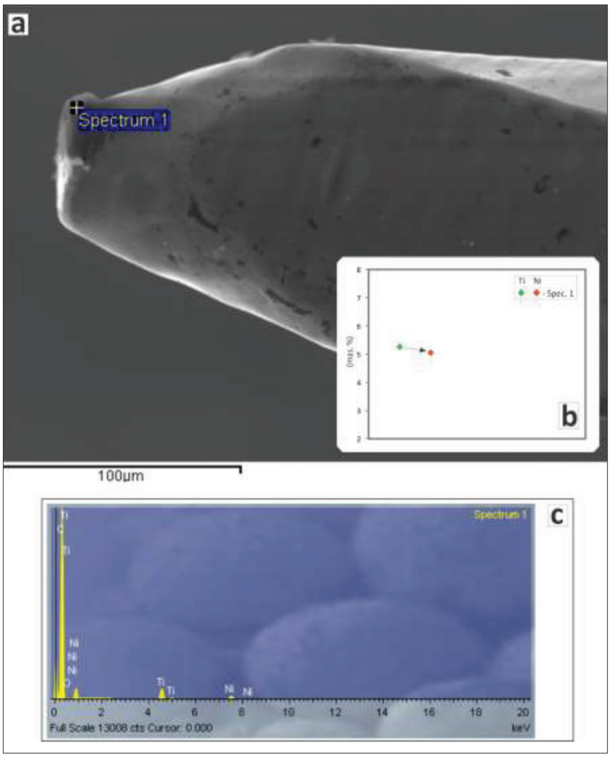

Figure 1. SEM-EDS analysis of the new ProTaper (Sx) instrument a) SE image of the tip of ProTaper (Sx) instrument with the marked point where EDS analysis was performed, parts of its surface, b) nickel and titanium contents (wt%), c) spectrum of the analysed point
Slika 1. SEM-EDS analiza površine novog ProTaper (Sx) instrumenta a) SE snimak vrha ProTaper (Sx) instrumenta sa označenom tačkom u kojoj je izvršena EDS analiza, dela njegove površine, b) sadržaji nikla i titanijuma (mas %), c) izgled spektra 1 za analizu u tački 1 (a)

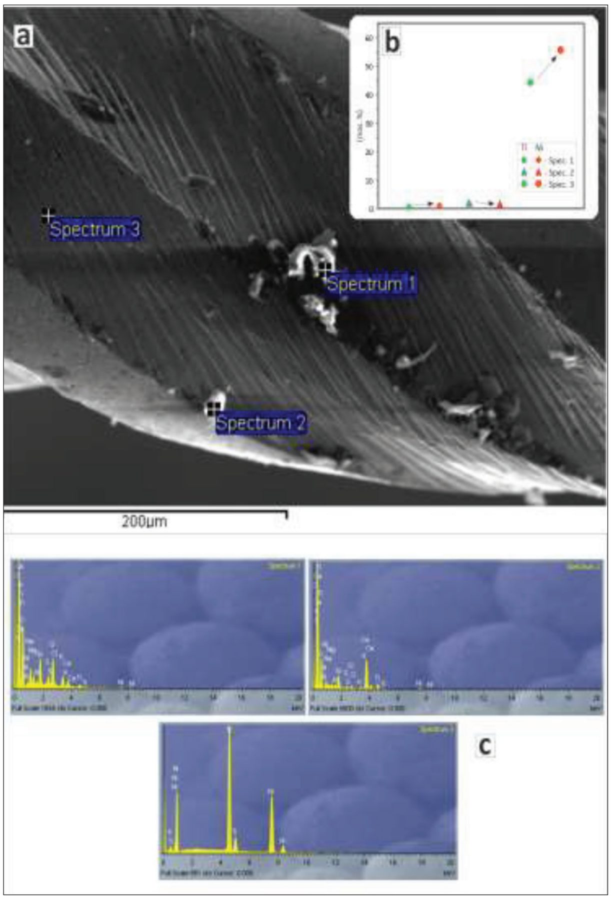

Figure 2. SEM-EDS analysis of a new K3 (25-0.10) instrument
a) SE image of K3 instrument surface with marked points where EDS analyzes were performed, b) nickel and titanium contents (wt%) at the analysed points (spectra 1–3),
c) spectrums of the analysed points 1–3
Slika 2. SEM-EDS analiza površine novog K3 (25-0.10) instrumenta a) SE snimak površine K3 instrumenta sa označenim tačkama u kojima su vršene EDS analize, b) sadržaji nikla i titanijuma (mas %) u tačkama analiziranja (spektri 1–3),
c) izgled spektara 1–3

Table 2. EDS analysis of the sample of ProTaper Universal (Sx) instrument in point 1 (Figure 1)
Tabela 2. SEM-EDS analiza uzorka vrha ProTaper Universal (Sx) instrumenta u tački 1 sa Slike 1

Spectrum	C	O	Ti	Ni	Total Ukupno
Spectrum 1	88.1	1.5	5.3	5.1	100.0

Table 3. EDS analysis of sample K3 (25-0.10) in points 1–3 (Figure 2)
Tabela 3. SEM-EDS analiza površine uzorka K3 (25-0.10) instrumenta u tačkama 1–3 (Slika 2)

Spectrum	C	O	Na	Mg	Al	Si	S	Cl	K	Ca	Ti	Ni	Total Ukupno
Spectrum 1	47.6	37.4	2.3	1.0	0.2	2.5	1.0	3.6	1.6	1.1	0.6	1.1	100.0
Spectrum 2	49.8	34.0	0.8	0.6	0.4	1.8	0.3	0.5	0.4	7.7	2.0	1.7	100.0
Spectrum 3	0.0	0.0	0.0	0.0	0.0	0.0	0,0	0.0	0.0	0.0	44.3	55.7	100.0

of K3 (27.43%) and ProTaper Universal group (27.21%). Occurrence of defects and impurities on new Ni-Ti instruments was recorded in Mtwo group in 20.43% and in HyFlex group in 17.2%. The lowest percentage of defects on the apical and middle surface was shown by BioRaCe sets (7.67%). Analyzing the results, a higher occurrence of defects on the apical surfaces of the working part of tested instruments can be noticed (53.49% on the apical and 46.51% on the middle third) (Table 4).

The presence of pitting and fritting (apical and middle third 100%), the presence of defects in the form of metal strips (apical third 83.33%, middle third 38.89%) and corrosion on only one instrument (apical and middle third) (Figure 3) were noticed on the surface of working part of new K3 instruments. Debris was observed in apical third in 77.8% of instruments and in middle third in 44.44% of K3 instruments.

SEM analysis of Mtwo instruments indicated the presence of metal strips in apical (50%) and middle third (22.2%). The corrosion was observed only on one instrument (the thinnest one) (10 / 0.4) in its middle third (Figure 4).

Blunt cutting edge of Mtwo instruments was observed on apical surface of one instrument (25 / 0.6) and defect in the form of disruption of cutting edge in middle third of

Table 4. Presence of defects and impurities on the working part of new Ni-Ti instruments
Tabela 4. Prisustvo defekata i nečistoća na radnom delu novih Ni-Ti instrumenata

		K3		*Mtwo*		*ProTaper Universal*		*HyFlex*		*BioRaCe*	
		Apical third % apikalna trećina %	**Middle third % srednja trećina %**	**Apical third % apikalna trećina %**	**Middle third % srednja trećina %**	**Apical third % apikalna trećina %**	**Middle third % srednja trećina %**	**Apical third % apikalna trećina %**	**Middle third % srednja trećina %**	**Apical third % apikalna trećina %**	**Middle third % srednja trećina %**
1	No Bez vidljivih defekata	0	0	0	0	0	0	0	0	0	0
2	Pitting Jamičasta udubljenja	100	100	0	0	83.3	88.8	0	0	0	0
3	Fretting Žljebovi	100	100	100	100	100	100	100	100	33.3	27.7
4	Microfractures Mikrofrakture	0	0	0	0	0	0	0	5.5%	0	0
5	Complete fractures Kompletne frakture	0	0	0	0	0	0	0	0	0	0
6	Metal flash Metalna uglačanost	0	0	0	0	0	0	0	0	0	0
7	Metal strips Metalni opiljci	83.3	38.8	50.0	22.2	50.0	38.8	5.5	0	50.0	38.8
8	Blunt cutting edge Zatupljene sečivne ivice	0	0	5.5	0	0	0	0	0	0	0
9	Disruption of cutting edges Prekid sečivne ivice	0	0	0	5.5	5.5	5.5	0	0	0	0
10	Corrosion Korozija	5.5	5.5	5.5	0	11.1	11.1	0	0	0	0
11	Debris Debris	77.7	44.4	100	100	100	55.5	100	100	11.1	22.2
	Σ	15.34	12.09	10.93	9.53	14.65	12.56	8.60	8.60	3.95	3.72
	Σ	27.43		20.46		27.21		17.2		7.67	

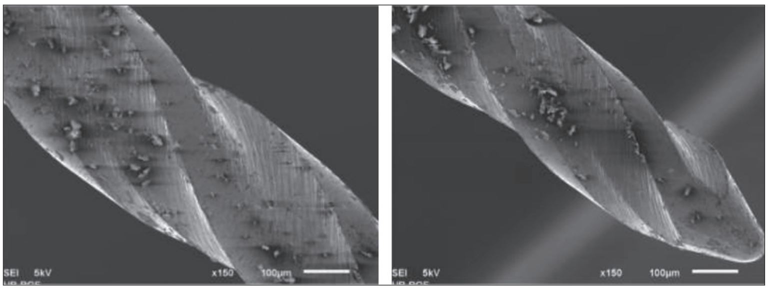

Figure 3. SE image of new K3 instruments: a) surface of middle third (25/0.10) with defects in the form of metal strips, pitting and fretting (×150) b) surface of apical third (25/0.08) where defects in the form of metal strips, pitting and fretting can be observed (×150).
Slika 3. SE snimak površine novih K3 instrumenata: a) površina srednje trećine (25/0,10) sa defektima u vidu metalnih opiljaka, žljebova i jamičastih udubljenja (×150); b) površina apikalne trećine (25/0,08), gde se uočava prisustvo defekta u vidu metalnih opiljaka, žljebova i jamičastih udubljenja (×150)

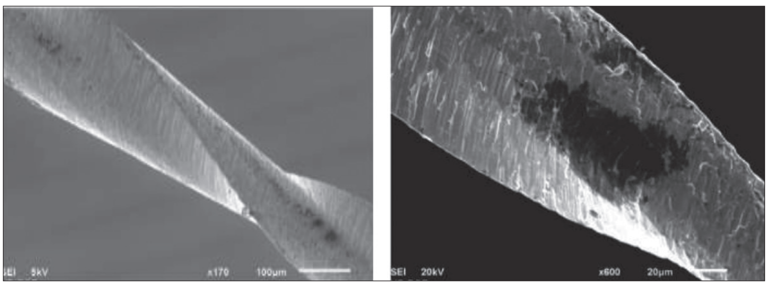

Figure 4. SE image of new Mtwo instrument: a) surface of middle third (10/0.4) with the presence of corrosion (×170); b) detail from the previous image at higher magnification (×600)
Slika 4. SE snimak novog Mtwo instrumenta: a) površina srednje trećine (10/0,4) sa prisustvom korozije (×170); b) detalj sa prethodnog snimka na većem uvećanju (×600)

the instrument (20 / 0.6) (Figure 5). Debris (confirmed by EDS analysis) was observed on all instruments, both in the apical and middle third (100%).

The most common defects on the working surface of new ProTaper Universal instruments were changes in the form of fretting (apical and middle third, 100%) and pitting (apical third 83.3% and middle third 88.8%) (Table 4, Figure 6). Metal strips were detected on the apical (50%) and middle third (38.8%). A defect on the cutting edge (disruption of its continuity) was observed on one, the most conical instrument (Sx) (apical and middle third), and corrosion on the apical (11.1%) and middle part (11.1%) (Figure 7). Contamination in the form of debris was noticed in apical third (100%) and in middle third of slightly more than a half (55.5%) of ProTaper Universal sets. The most common defects on new HyFlex instruments were the appearance of fretting in the form of debris on apical and middle segments of all instruments (100%) (Figure 8). A defect in the form of a microfracture was observed on the apical part of the instrument (25-0.08) as well as the appearance of metal strips (25-0.04).

The results of SEM analysis of BioRaCe sets show the most frequent occurrence of metal strips (apical 50% and middle third

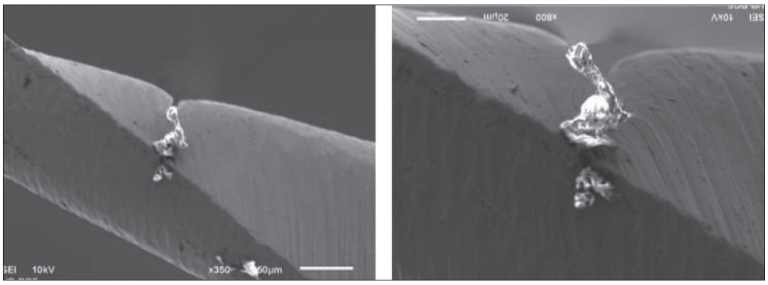

Figure 5. SE image of new Mtwo instrument (20/0.6): a) area of middle third with a disruption of cutting edge defect (×350); b) detail from the previous image at higher magnification (×800)
Slika 5. SE snimak novog Mtwo instrumenta (20/0,6): a) površina srednje trećine na kojoj se uočava prisusutvo defekta u vidu prekida sečivne ivice (×350); b) detalj sa prethodnog snimka na većem uvećanju (×800)

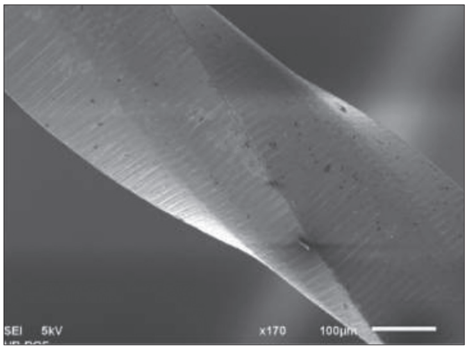

Figure 6. SE image of middle part of ProTaper Universal instrument (F2) with pitting and fretting (×170)
Slika 6. SE snimak srednjeg dela ProTaper Universal instrumenta (F2) sa jamičastim udubljenjima i žljebovima (×170)

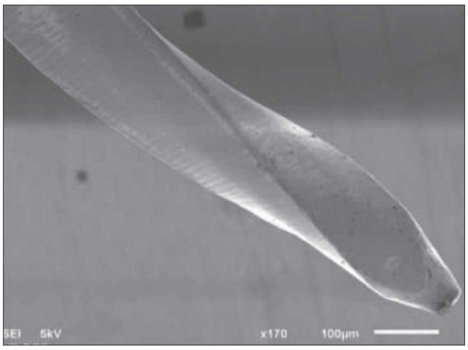

Figure 7. SE image of apical third of new ProTaper instrument (Sx) with corrosion and discontinuity of cutting edge (apart from the presence of pitting and fretting) (×170)
Slika 7. SE snimak apikalne trećine novog ProTaper instrumenta (Sx) sa korozijom i prekidom kontinuiteta (pored prisustva jamičastih udubljenja i žljebova) (×170)

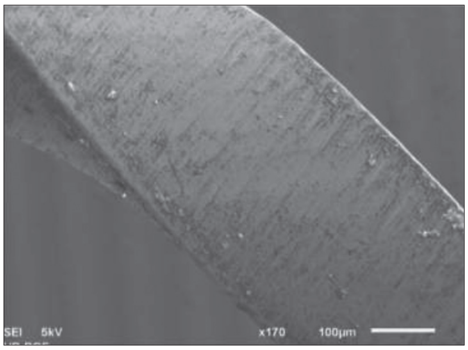

Figure 8. SE image of the middle third surface new HyFlex instrument (30-0.04) with the presence of fretting and debris (×170)
Slika 8. SE snimak površine srednje trećine novog HyFlex instrumenta (30-0.04) sa prisusutvom žljebova i debrisa (×170)

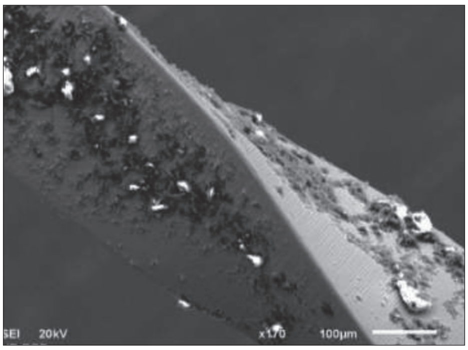

Figure 9. SE image of the surface of new BioRaCe instrument (No. 3) i.e. its middle third with the presence of metal strips and fretting (×170)
Slika 9. SE snimak površine srednje trećine novog BioRaCe instrumenta (br. 3) sa prisusutvom metalnih opiljaka i žljebova (×170)

38.8%) and fretting (apical 33.3% and middle third 27.7%) (Table 4, Figure 9). Debris contamination detected by EDS analysis was observed on the apical (11.1%) and middle surface (22.2%) of the instruments.

A statistically significant difference in the presence of fretting was observed between K3, MTwo, ProTaper and Hyflex instruments between the apical (by $p < 0.05$) and middle third (by $p < 0.05$).

Significant differences related to the presence of metal strips were observed between K3 and HyFlex instruments (by $p < 0.05$), between ProTaper Universal and HyFlex (by $p < 0.05$), MTwo and HyFlex (by $p < 0.05$) and BioRaCe and HyFlex instruments (for $p < 0.05$)). The difference was also significant in the occurrence of metal strips in apical third between K3 and BioRaCe group of instruments (by $p < 0.05$) between K3 and MTwo (by $p <0.05$) and K3 and ProTaper group, respectively (by $p < 0.05$).

In the middle third, a statistically significant difference in the occurrence of metal strips was observed between the HyFlex group and K3, BioRaCe, ProTaperUniversal and MTwo instruments (by $p < 0.05$). The difference was also significant between the apical and middle third of K3 instruments (by $p < 0.05$).

The difference was also significant in the values of debris in the apical segment between K3 and ProTaper Universal group (for $p < 0.05$), K3 and MTwo group (for $p < 0.05$), and between K3 and HyFlex group of instruments (for $p < 0.05$). A statistically significant difference was also observed between K3 and BioRaCe group (for $p < 0.05$), ProTaperUniversal and BioRaCe group (for $p < 0.05$), MTwo and BioRaCe group (for $p < 0.05$), respectively, HyFlex and BioRaCe groups (for $p < 0.05$). In the middle third, the difference was significant between K3 and MTwo (for $p < 0.05$) and K3 and HyFlex instruments (for $p < 0.05$). In the ProTaper Universal group, a statistically significant difference in the occurrence of debris was observed between the apical and middle third (by $p < 0.05$).

DISCUSSION

SEM analysis of various surface irregularities, manufacturing defects and contamination of new Ni-Ti rotating instruments showed that there is not a single tested instrument without defects or impurities on the working surface. In this study, the presence of various defects and impurities was found in all new Ni-Ti instruments (five different commercial sets), with a slightly higher prevalence observed in their apical third. Although there is confirmation of their sterility on the factory packaging, the presence of defects and dirt on the active part of new Ni-Ti instruments is a proven reality, which is documented by the results of various studies [8, 10–19]. The complicated process of machining the initial Ni-Ti wire often causes the occurrence of surface deformations and cracks due to the traces of milling and machining, but also the appearance of polished surfaces on the cutting edges of instruments [5, 10].

Changes on the surface can compromise blade efficiency of instruments and become sites for potential corrosion. Also, these points represent the sites of initiation of defects, contributing to degradation of mechanical properties and occurrence of micro or complete fractures during their clinical use [5, 7, 13]. Arens et al. presented an interesting study on the incidence of fractures after the first use of new Ni-Ti instruments (0.9%), while Shen cites inadequate manipulation and existence of manufacturing defects as the cause of this complication [12, 13].

Due to the higher forces and speed that are necessary for the processing of Ni-Ti alloys, it is possible to cause burning sawdust and formation of hardened places. These are the hardened parts that are more difficult to process, and they represent the zones with higher probability of deformation and fractures [16].

The manner in which defects are formed during the formation of Anusavica and Phillips alloys has been attributed to the specific phase transformation and recrystallization of Ni-Ti alloy [20]. Recrystallization represents the change in the type of lattice depending on the temperature (e.g. titanium at 882 ° C changes from a hexagonal to a monoclinic structure), where the rate of crystallization affects the regularity of crystal structure [20].

The most common type of surface irregularities on working surface of new instruments in this study was the appearance of fretting. Clinical significance of fretting is potentially increased possibility of its screwing (due to the friction that is caused by uneven surface) and increased incidence of fracture [13].

Presence of metal strips as a consequence of the production process was observed on the work surface of all tested instruments. The correlation between the high prevalence of metal strips and the higher conicity of K3 instruments in this study (conicity greater than .06) is in accordance with the results of Marending et al. who indicated that metal strips are formed as a result of the production process of Ni-Ti instruments [11]. Using SEM analysis, Van Eldik et al. noticed the presence of a large amount of metal strips on the surface of new Ni-Ti instruments, immediately after opening the original packaging [14]. This type of contamination leads to a decrease in cutting efficiency, and metal strips can be retained in the dentinal walls of the canal or in the periapical tissue during instrumentation. Van Eldik proved that possible contamination of periapical tissue with these metal strips could reduce the course of tissue repair and compromise the success of endodontic therapy [14]. According to the results of Stefanescu et al. metal particles can be transported during instrumentation and active irrigation through the apical foramen and cause an allergic reaction of the periapical tissue [8]. It has been shown that metal ions as potential hapten allergens can cause type 1 reaction, with a possible immediate or delayed dermal or mucosal reaction. Allergic reactions in endodontics are extremely rare, but the consequences of allergic reactions such as symptoms of delayed apical healing, persistent discomfort after canal obstruction, can increase their number significantly [8].

The presence of debris was also observed on the working surfaces of all types of tested Ni-Ti instruments. Titanium alloys are difficult to machine due to their elasticity and require higher cutting forces compared to steel. Ni-Ti alloys are intensively glued to the tool with which they are processed, so the protection of materials is achieved by oxidizing the surface or metal coating, which are removed chemically after processing, but may still remain on their surface [16]. Electropolishing the surface of BioRaCe instruments increases cutting efficiency, while reducing defects in the production process and possible debris contamination [21]. The significant frequency of debris in the apical third of K3 and ProTaper Universal instruments in relation to their middle third confirms higher contamination of the apical segment due to the more complex production of thinner apical part. This finding is consistent with studies by Eggert and Alapati, which indicated a higher incidence of debris in the apical segment of new Ni-Ti instruments [10, 22].

Working surface defects in the form of pitting were observed only in two groups of new instruments, but in a high percentage (K3 and ProTaper Universal). The appearance of pitting occurs during the production process, as during melting of elemental nickel and titanium, the rates of their mutual diffusion during heating differ, which leads to the formation of void spaces [23]. Nickel atoms diffuse faster into titanium than titanium atoms in the opposite direction. Thus, the mass transport is not balanced which can lead to the formation of void spaces in the nickel after alloying. These cavities are known as the Kirkendal porosity or Kirkendal effect [23].

Nagumo presented the evidence on the significance that these defects have on mechanical characteristics of Ni-Ti instruments, as well as the exact mechanism of their influence [24]. He observed that alloy could absorb hydrogen from saliva and form hydride bonds with Ni-Ti lattice atoms that are stable at room temperature. This change in the molecular structure leads to the change in physical properties of the alloy, causing hydrogen porosity. Asaoka also pointed out that diffusion of hydrogen through a Ni-Ti alloy forms hydride phases on the surface of a material that has a more brittle structure [25]. This newly formed hydride layer on the active surface of Ni-Ti instrument is of different thickness thus causing microcracks during clinical work. By providing an absolutely dry working field, this mechanism is not important, but it can have an impact during the process of cleaning and sterilization of instruments, when the instruments are exposed to a longer action of ionizing liquids [25].

Corrosion of the working part of Ni-Ti instruments was not observed on HyFlex and BioRaCe instruments, and in other groups it was observed in a small percentage. The low degree of corrosion on Ni-Ti instruments confirms the resistance of this alloy to corrosion, but also the non-exposure of new instruments to corrosive factors [26].

Findings of defects on the surface of new Ni-Ti instruments in the form of blunting of the cutting edge, disruption of the blade edge and microfracture, only confirm the problems of their production. Microfractures on new instruments are, according to research by Marending and Barbakow, the result of manufacturing process of larger and more conical but less flexible instruments [11]. According to the most researchers, cracks or microfractures are the most dangerous defects that a file can have [24, 25]. If instruments with this defect are activated in the canal, during rotation and screwing, they break immediately. Microfracture affects high sensitivity of the instrument to the accumulation of cyclic fatigue and inevitable fractures [11, 24, 25].

Subsequent heat treatment of finished Ni-Ti instruments (HyFlex) potentially offers the most promising method of manufacturing rotating instruments [27]. These instruments do not have the shape memory that traditional Ni-Ti instruments have, and a special thermomechanical procedure significantly increases their flexibility [28]. The research results in this study show the lowest contamination of Hyflex system with metal strips. The low prevalence of this contamination can be explained by their specific heat treatment that reduces irregularities on their surface. Heat treatment, apart from the change in microstructure (increased flexibility), also leads to the appearance of a cleaner and more regular surface of these instruments [28, 29].

Following the results of our study, a significantly lower prevalence of defects in BioRaCe group is observed. This finding is in accordance with the results of a research on a significant reduction of surface irregularities of electropolished instruments [3]. Electropolishing creates a homogeneous oxide layer during the production process, which reduces the appearance of surface defects and increases resistance to corrosion and fracture [27]. Electropolished surface of instruments is visibly brighter than the untreated surface [26]. By introducing current through the solution, a thin passive layer is formed and the surface dissolves into the electrolyte, which also leads to the selective removal of surface defects [4].

In order to improve microstructure of the working surface of Ni-Ti instruments and improve mechanical properties, flexibility, fatigue resistance, i.e. cutting efficiency, manufacturers have used various techniques in recent years (ionic application, plasma immersion, titanium oxide coating, thermal nitriding, thermal treatments and cryogenic treatments, electropolishing) [3, 26, 28, 30].

CONCLUSION

Based on the results of this study, it can be concluded that production defects or impurities (one or more) were observed on all new tested instruments. The most common type of irregularity was the existence of fretting, debris and metal strips on the working part of instruments. No organic debris was observed on electropolished surface of BioRaCe instruments. The results of our study indicate that cleaning and sterilization of instruments before the first use is mandatory. However, further research is needed in order to start manufacturing instruments without defects and impurities.

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Karakteristike površine novih rotirajućih nikl-titanijumskih instrumenata – SEM-EDS analiza

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KRATAK SADRŽAJ

Uvod Moderna endodontska procedura podrazumeva upotrebu mašinskih rotirajućih Ni-Ti instrumenata tokom hemomehaničke obrade kanala korena.

Cilj ovog rada je bio da se primenom SEM-EDS metode analiziraju površine novih (neupotrebljenih) rotirajućih endodontskih instrumenata i utvrdi učestalost pojave proizvodnih defekata ili nečistoća na njihovim radnim površinama.

Materijal i metoda U istraživanje je uključeno pet različitih novih setova mašinskih endodontskih Ni-Ti instrumenata: K3, Mtwo, ProTaper Universal, HyFlex i BioRaCe. Radni deo svakog instrumenta je podvrgnut SEM-EDS analizi (uvećanja ×150 do ×2000), čime su utvrđene morfološke karakteristike površine instrumenata i hemijski sastav zatečenih nečistoća. Statistička analiza je urađena primenom Fišer testa ($p < 0,05$).

Rezultati Rezultati SEM-EDS analize su pokazali da ne postoji nijedan novi instrument bez defekta na svojoj površini. Najučestaliji defekti su uočeni kod K3 (27,43%) i ProTaper Universal grupe (27,21%), a najmanje ih je bilo na BioRaCe instrumentima (7,67%). Najzastupljeniji tip defekata kod testiranih instrumenata je bilo prisustvo žljebova. Osim toga, utvrđeno je i prisustvo debrisa i metalnih opiljaka na svim instrumentima, dok je korozija radnog dela primećena samo kod K3, ProTaper Universal i Mtwo sistema u malom procentu.

Zaključak Na osnovu rezultata ovog istraživanja može se zaključiti da su na svim ispitivanim instrumentima uočeni proizvodni defekti, od kojih su najzastupljeniji žljebovi. Takođe su registrovane i nečistoće kao što su debrisa i metalni opiljci. Na elektropoliranoj površini BioRaCe instrumenata nije uočeno prisustvo organskog debrisa, ali je registrovan mali procenat ostalih tipova defekata.

Ključne reči: Ni-Ti instrumenti; proizvodni defekti; SEM-EDS

UVOD

Primena mašinskih nikl-titanijumskih (Ni-Ti) instrumenata sa svojstvima pamćenja oblika, izrazite biokompatibilnosti i otpornosti na koroziju, uvela je endodontsku proceduru u novu eru. Rotirajući Ni-Ti instrumenti su omogućili bržu i efikasnu preparaciju, odnosno smanjenu mogućnost pojave proceduralnih grešaka tokom čišćenja i oblikovanja kanala različite kanalske morfologije [1]. Brojne inovacije u dizajnu instrumenata, tokom poslednjih godina vezane za površinsku i termičku obradu Ni-Ti legure, uticale su na efikasnost i neophodnu sigurnost tokom endodontskog tretmana [2, 3, 4].

U izradi endodontskih instrumenata koristi se Ni-Ti legura u odnosu 56 : 44 = Ti : Ni, čime se postiže njihov ekviatomski odnos. Iako je samo jedan proizvođač (Dentsply, Maillefer Instruments SA, Ballaigues, Švajcarska) objavio apsolutni sastav i detaljan tehnološki proces njihove izrade, pretpostavlja se da je to najbolji odnos koji leguri daje superelastična svojstva [5].

Proizvodnja Ni-Ti mašinskih endodontskih instrumenata je mnogo komplikovanija u poređenju sa procesom izrade čeličnih instrumenata koji se izrađuju hladnim uvijanjem prethodno profilisanih žičanih konusa [2]. Ni-Ti instrumenti nastaju specifičnim brušenjem, odnosno urezivanjem određenog profila u centralno stablo Ni-Ti žice [5]. Novije tehnike proizvodnje uključuju kombinaciju termičke obrade legure i istovremenog uvijanja, radi veće fleksibilnosti i bolje otpornosti na torziju i ciklični zamor [3]. Iako se u procesu izrade komplikovanog dizajna Ni-Ti instrumenata koristi savremena kompjuterska tehnologija, često se javljaju površinski nedostaci u vidu žljebova, jamičastih udubljenja, pukotina i nečistoća koji mogu povećati vulnerabilnost na frakturu [6]. Uočeno je da defekti na površini deluju kao tačke koncentracije napona, dovode do inicijacije i širenja pukotina, odnosno čestih pojava frakture tokom aktivacije instrumenta [7].

Na površini novih endodontskih instrumenata se mogu naći i različiti metalni ostaci i nečistoće organskog i neorganskog porekla. Tokom instrumentacije ovi metalni opiljci se mogu inkorporirati u dentinski zid ili potisnuti u periapeksno tkivo i izazvati alergijsku reakciju [8]. Upotreba instrumenata na kojima su se zatekle organske nečistoće nosi rizik i od moguće unakrsne infekcije [9].

Cilj ovog rada je bio da se primenom skenirajuće elektronske mikroskopije sa energetsko-disperzivnom spektrometrijom (SEM-EDS) analiziraju površine novih rotirajućih endodontskih instrumenata i utvrdi učestalost pojave proizvodnih defekata ili nečistoća na njihovim radnim površinama.

MATERIJAL I METOD

U istraživanju su korišćena po tri osnovna seta (svaki set po šest instrumenata) pet različitih sistema mašinskih endodontskih Ni-Ti instrumenata: K3 (SybronEndo Co, USA), Mtwo (VDW, Munich, Germany), ProTaper Universal (Dentsply Maillefer, Switzerland), HyFlex (Coltene Whaledent gruppe, Switzerland) i BioRaCe (FKG DENTAIRE Swiss Dental Produkts, Switzerland) (Tabela 1).

Skenirajuća elektronska mikroskopija sa energetsko-disperzivnom spektrometrijom (SEM-EDS) realizovana je u Laboratoriji za SEM Rudarsko-geološkog fakulteta Univerziteta u Beogradu, na SEM-u tipa JEOL JSM-6610LV, Japan. Instrumenti su analizirani bez pripreme (direktno iz fabričkog pakovanja). Izrađeni su snimci pomoću detektora za sekundarne elektrone (SE snimci – *second electron*) na uvećanjima u rasponu od 150 do 2000 puta. Hemijska analiza je urađena na nepoliranim uzorcima pomoću EDS detektora (tip X-Max Large Area Analytical Silicon Drifted spectrometer, Oxford Instruments) uz upotrebu unutrašnjih standarda. Dobijeni hemijski sastav je predstavljen kao sadržaj hemijskih elemenata u masenim procentima (wt%), normalizovan na 100%. Granica detekcije je za većinu elemenata iznosila oko 0,1 wt%. Ovako urađena hemijska analiza se smatra semikvantitatvnom, jer je urađena na nepoliranim površinama.

Ukupno je napravljeno 540 snimaka apeksne i srednje trećine instrumenata iz dva različita pravca. Za svaku površinu instrumenta napravljena su po tri SE snimka. Snimke su pregledala dva istraživača, a usaglašavanje njihovih rezultata izvršeno je analizom Cohen Kappa.

Primenjena je kvalitativna analiza prisustva različitih nepravilnosti i grešaka na radnoj površini Ni-Ti instrumenata u skladu sa preporukama Kristine Egert i sar. [10]. Na instrumentima je registrovano prisustvo: jamičastih udubljenja, žljebova, mikrofraktura, fraktura, metalne uglačanosti, metalnih opiljaka, tupih sečivnih ivica, korozije, debrija i uočen je prekid sečivnih ivica.

Statistička analiza dobijenih rezultata je urađena primenom Fišerovog testa ($p < 0,05$).

REZULTATI

Rezultati SEM-EDS analize novih Ni-Ti setova prikazani su u tabelama 2, 3 i 4 i na slikama 1–9.

Analizom SE snimaka utvrđeno je postojanje kontaminacije na površini radnog dela ispitivanih instrumenata, a naknadnom EDS analizom je utvrđen njen hemijski sastav. Na taj način je urađena podela na instrumente kontaminirane debrisom i kontaminirane metalnim opiljcima. Primeri SEM-EDS analize sa izgledom izvršenog spektra su dati na slikama 1 i 2.

EDS analiza u tački 1 (spektar 1, Slika 1, Tabela 2) pokazuje da je dominantni element u uzorku ugljenik (88,1 mas%) sa malim sadržajem kiseonika (1,5 mas%), te je nečistoća na ovom ProTaper Universal instrumentu okarakterisana kao debris organskog porekla. Sadržaji nikla i titanijuma (spektar 1) odražavaju sastav instrumenta.

Na osnovu EDS analize nečistoća na ovom K3 instrumentu je okarakterisana kao kombinacija debrisa organskog porekla i kontaminacija metalnim opiljcima.

Pojava nikla i titanijuma u analizi 1 (spektar 1, Slika 2) i nešto više u analizi 2 (spektar 2, Slika 2), kao i u prethodnom slučaju, predstavlja distribuciju ovih elemenata u strukturi samih instrumenata.

Rezultati SEM-EDS analize ukazuju na najučestaliju pojavu defekata i nečistoća u sistemima K3 (27,43%) i ProTaper Universal grupe (27,21%). Pojava defekata i nečistoća na novim Ni-Ti instrumentima je u Mtwo grupi zabeležena u 20,43%, a u HyFlex grupi u 17,2%. Najmanji procenat zastupljenosti defekata na apikalnoj i srednjoj površini pokazali su BioRaCe setovi (7,67%). Analizirajući rezultate, uočava se veća pojava defekata na apikalnim površinama radnog dela testiranih instrumenata (53,49% na apikalnoj i 46,51% na srednjoj trećini) (Tabela 4).

Na površini radnog dela novih K3 instrumenata uočeno je prisustvo jamičastih udubljenja i žljebova (apikalna i srednja trećina 100%), prisustvo defekta u vidu metalnih opiljaka (apikalna trećina 83,33%, srednja trećina 38,89%) i pojava korozije samo na jednom instrumentu (apikalna i srednja trećina) (Slika 3). Debris je uočen na apikalnoj trećini kod 77,8% instrumenata i na srednjoj trećini kod 44,44% K3 instrumenata.

SEM-EDS analiza Mtwo instrumenata je ukazala na prisustvo metalnih opiljaka na apikalnoj (50%) i na srednjoj trećini (22,2%). Defekt u vidu korozije je primećen samo na jednom (najtanjem) instrumentu (10/0,4), u njegovoj srednjoj trećini (Slika 4).

Zatupljenost sečivne ivice Mtwo instrumenata je uočena na apikalnoj površini jednog instrumenta (25/0,6) a defekt u vidu prekida sečivne ivice na srednjoj trećini instrumenta (20/0,6) (Slika 5). Debris (koji je potvrđen EDS analizom) uočen je na svim instrumentima, i u apikalnoj i srednjoj trećini (100%).

Najzastupljeniji defekti na površini radnog dela novih ProTaper Universal instrumenata bile su promene u vidu žljebova (apikalna i srednja trećina, 100%) i jamičastih udubljenja (apikalna trećina 83,3% i srednja trećina 88,8%) (Tabela 4, Slika 6). Metalni opiljci su detektovani na apikalnoj (50%) i srednjoj trećini (38,8%). Defekt na sečivnoj ivici (prekid njenog kontinuiteta) uočen je na jednom, najkoničnijem instrumentu (Sx) (apikalna i sredna trećina), a korozija na apikalnom (11,1%) i srednjem delu (11,1%) (Slika 7). Kontaminacija u vidu debrisa je bila zastupljena na apikalnoj trećini (100%) i u nešto više od polovine (55,5%) u srednjoj trećini ProTaper Universal setova.

Najzastupljeniji defekt na novim HyFlex instrumentima je bila pojava žljebova kao prisustvo nečistoće u vidu debrisa na apikalnim i srednjim segmentima svih instrumenata (100%) (Slika 8).

Defekt u vidu mikrofrakture je uočen na apikalnom delu instrumenta (25-0,08), kao i pojava metalnih opiljaka (25-0,04).

Rezultati SEM analize BioRaCe setova pokazuju najučestaliju pojavu metalnih opiljaka (apikalna 50% i srednja trećina 38,8%) i žljebova (apikalna 33,3% i srednja trećina 27,7%) (Tabela 4, Slika 9). Kontaminacija debrijem detektovana EDS analizom je uočena na apikalnoj (11,1%) i srednjoj površini (22,2%) instrumenata.

Statistički značajna razlika u prisustvu žljebova je uočena između intrumenata K3, MTwo, ProTaper i Hyflex između apikalne (za $p < 0,05$) i srednje trećine (za $p < 0,05$).

Značajne razlike vezane za prisusutvo metalnih opiljaka uočene su između K3 i HyFlex instrumenata (za $p < 0,05$), između ProTaper Universal i HyFlex (za $p < 0,05$), MTwo i HyFlex (za $p < 0,05$) i BioRaCe i HyFlex instrumenata (za $p < 0,05$). Razlika je bila značajna i u pojavi metalnih opiljaka u apikalnoj trećini između K3 i BioRaCe grupe instrumenata (za $p < 0,05$), između K3 i MTwo (za $p < 0,05$), odnosno K3 i ProTaper grupe (za $p < 0,05$).

U srednjoj trećini statistički značajna razlika u pojavi metalnih opiljaka uočena je između HyFlex grupe i K3, BioRaCe, ProTaperUniversal i MTwo intrumenata (za $p < 0,05$). Razlika je bila značajna i između apikalne i srednje trećine instrumenata K3 (za $p < 0,05$).

Razlika je bila značajna i u vrednostima pojave debrisa u apikalnom segmentu između K3 i ProTaper Universal grupe (za $p < 0,05$), K3 i MTwo grupe (za $p < 0,05$), odnosno između K3 i HyFlex grupe instrumenata (za $p < 0,05$). Statistički značajna razlika je uočena i između K3 i BioRaCe grupe (za $p < 0,05$), ProTaperUniversal i BioRaCe grupe (za $p < 0,05$), MTwo i BioRaCe grupe (za $p < 0,05$), odnosno HyFlex i BioRaCe grupe (za $p < 0,05$). U srednjoj trećini razlika je bila značajna između K3 i MTwo (za $p < 0,05$) i K3 i HyFlex instrumenata (za $p < 0,05$). U ProTaper Universal grupi statistički značajna razlika u pojavi debrisa uočena je između apikalne i srednje trećine (za $p < 0,05$).

DISKUSIJA

SEM analiza prisustva različitih nepravilnosti površine, proizvodnih grešaka i kontaminacija novih Ni-Ti rotirajućih instrumenata pokazala je da ne postoji nijedan ispitivani instrument bez defekta ili nečistoća na radnoj površini. U ovoj studiji je utvrđeno prisustvo različitih defekata i nečistoća kod svih novih Ni-Ti instrumenata (pet različitih komercijalnih setova), pri čemu je nešto veća zastupljenost uočena u njihovoj apikalnoj trećini. Iako na fabričkom pakovanju postoji potvrda o njihovoj sterilnosti, prisustvo defekata i prljavštine na aktivnom delu novih Ni-Ti instrumentima je dokazana realnost, što je dokumentovano rezultatima različitih studija [8, 10–19].

Komplikovani proces mašinske obrade početne Ni-Ti žice često uzrokuje nastanak površinskih deformacija i pukotina usled tragova glodanja i obrade, ali i pojavu uglačanih površina na sečivnim ivicama instrumenata [5, 10].

Površinske promene mogu kompromitovati sečivnu efikasnost instrumenata i postati mesta za mogući nastanak korozije. Takođe, ove tačke predstavljaju mesta inicijacije defekata, doprinoseći degradaciji mehaničkih svojstava, i pojavi mikro ili komletne frakture tokom njihove kliničke upotrebe [5, 7, 13]. Arens i saradnici su prezentovali zanimljivu studiju o učestalosti frakture posle prve upotrebe novih Ni-Ti instrumenata (0,9%), dok Shen kao uzrok ove komplikacije navodi neadekvatnu manipulaciju i postojanje proizvodnih defekata [12, 13].

Usled većih sila i veće brzine koje su neophodne za obradu Ni-Ti legure, postoji mogućnost paljenja strugotine i formiranja otvrdnutih mesta. To su delovi veće tvrdoće koji se teže obrađuju, te predstavljaju zone sa većom verovatnoćom za pojavu deformacija i fraktura [16].

Način na koji dolazi do formiranja defekata tokom nastanka legure Anusavice i Phillips su pripisali karakterističnoj faznoj transformaciji i prekristalizaciji Ni-Ti legure [20]. Prekristalizacija je pojava promene tipa rešetke u zavisnosti od temperature (npr. titan pri 882°C prelazi iz heksagonalne u monokliničnu strukturu), pri čemu brzina kristalizacije utiče na pravilnost strukture kristala [20].

Najučestaliji tip površinskih iregularnosti na radnoj površini novih instrumenata u ovoj studiji je bila **pojava žljebova**. Klinički značaj pojave žljebova je u povećanju mogućnosti njegovog ušrafljivanja (usled trenja koje postoji zbog neravne površine) i povećane incidence loma [13].

Prisustvo metalnih opiljaka kao posledica proizvodnog procesa zapaženo je na radnoj površini svih ispitivanih instrumenata. Korelacija između velike zastupljenosti metalnih opiljaka i veće koničnosti instrumenata K3 u ovoj studiji (koničnost veća od 0,06) u saglasnosti je sa rezultatima Marendinga i saradnika, koji su ukazali da metalni opiljci nastaju kao rezultat proizvodnog procesa i to češće kod debljih i koničnijih instrumenata [11].

Primenom SEM analize Van Eldik je sa saradnicima uočio prisusutvo velike količine metalnih opiljaka na površini novih Ni-Ti instrumenata, neposredno po otvaranju iz originalnog pakovanja [14]. Ovaj tip kontaminacije dovodi do smanjenja sečivne efikasnosti, a metalni opiljci se tokom instrumentacije mogu zadržati u dentinskim zidovima kanala ili u periapeksnom tkivu. Van Eldik je dokazao da moguća kontaminacija periapeksnog tkiva ovim metalnim opiljcima može redukovati tok reparacije tkiva i kompromitovati uspeh endodontske terapije [14]. Stefanesku i saradnici u svojim rezultatima dokazuju da čestice metala mogu biti transportovane tokom instrumentacije i aktivne irigacije kroz apeksni foramen i uzrokovati alergijsku reakciju periapeksnog tkiva [8]. Dokazano je da joni metala kao potencijalni haptenski alergeni mogu uzrokovati reakcije tipa 1, uz moguću trenutnu ili odloženu dermalnu ili sluzokožnu reakciju. Alergijske reakcije u endodonciji izuzetno su retke, ali posledice alergijskih reakcija, kao što su simptomi odloženog apikalnog zarastanja, uporne nelagodnosti nakon opturacije kanala, mogu značajno povećati njihov broj [8].

Prisustvo debrisa je, takođe, uočeno na radnim površinama svih tipova ispitivanih Ni-Ti instrumenata. Titanijumske legure se teško obrađuju zbog svoje elastičnosti, te zahtevaju veće sile rezanja u odnosu na čelik. Ni-Ti legura se intenzivno lepi za alat kojim se obrađuje, pa se zaštita materijala postiže oksidiranjem površine ili metalnim premazima, koji se nakon obrade uklanjaju hemijskim putem, koji može zaostati na njihovoj površini [16]. Elektropoliranjem površine instrumenata BioRaCe povećava se sečivna efikasnost, a istovremeno smanjuju defekti nastali u proizvodnom procesu i smanjuje mogućnost kontaminacije debrisom [21]. Značajnija učestalost pojave debrisa u apikalnoj trećini K3 i ProTaper Universal instrumenata u odnosu na njihovu srednju trećinu potvrđuje veću kontaminiranost apikalnog segmenta zbog kompleksnije izrade gracilnijeg apikalnog dela. Ovaj nalaz je u saglasnosti sa studijama koje su sproveli Eggert i Alapati, a koje su ukazale na veću pojavu debrisa u apikalnom segmentu novih Ni-Ti instrumenata [10, 22].

Defekti radne površine u vidu jamičastih udubljenja uočeni su samo kod dve grupe novih instrumenata ali u visokom procentu (K3 i ProTaper Universal). Pojava jamičastih šupljina nastaje tokom proizvodnog procesa, jer se tokom topljenja elementarnog nikla i titanijuma razlikuju brzine njihove međusobne difuzije pri zagrevanju, što dovodi do formiranja praznina [23]. Atomi nikla difunduju brže u titan nego atomi titanijuma u obrnutom smeru. Dakle, maseni transport nije izbalansiran, što može dovesti do stvaranja praznina u niklu nakon legiranja. Ove šupljine su poznate pod nazivom Kirkendalova poroznost ili Kirkendalov efekat [23].

Nagumo je izneo dokaze o značaju ovih defekata na mehaničke karakteristike Ni-Ti instrumenata, kao i tačnom mehanizmu njihovog uticaja [24]. On je zapazio da legura može da apsorbuje vodonik iz pljuvačke i formira hidridne veze sa atomima Ni-Ti rešetke koje su stabilne na sobnoj temperaturi. Ovakva promena molekularne strukture dovodi do promene fizičkih svojstava legure izazivajući hidrogensku poroznost. Asaoka je, takođe, ukazao da difuzija vodonika kroz Ni-Ti leguru formira faze hidrida na površini materijala koji ima krtiju strukturu [25]. Ovaj novonastali hidridni sloj na aktivnoj površini Ni-Ti instrumenta je različite debljine i tokom kliničkog rada dolazi do formiranja mikropukotina. Obezbeđenjem apsolutno suvog radnog polja, ovaj mehanizam nema značaja, ali može imati uticaja tokom procesa čišćenja i sterilizacije instrumenata, kada su instrumenti izloženi dužem dejstvu jonizujućih tečnosti [25].

Korozija radnog dela Ni-Ti instrumenata nije primećena na HyFlex i BioRaCe instrumentima, a i u ostalim grupama je zapažena u malom procentu. Mali stepen zastupljenosti korozije na Ni-Ti instrumentima potvrđuje otpornost ove legure na koroziju, ali i neizlaganje novih instumenata korozivnim faktorima [26].

Nalazi defekata na površini novih Ni-Ti instrumenata u vidu **zatupljenosti sečivne ivice**, **prekida sečivne ivice i mikrofrakture** samo potvrđuju problematiku njihove proizvodnje. Mikrofrakture na novim instrumentima su, prema istraživanjima Marendinga i Barbakowa, rezultat proizvodnog procesa većih i koničnijih, manje fleksibilnih instrumenata [11]. Pukotine ili mikrofrakture su po većini istraživača najopasniji defekti koji turpija može imati [24, 25]. Ukoliko se instrumenti sa ovim defektom aktiviraju u kanalu, prilikom rotacije i ušrafljivanja, oni se lome odmah. Mikropukotina utiče na veliku osetljivost instrumenta na akumulaciju cikličnog zamora i neminovne frakture [11, 24, 25].

Naknadna termička obrada gotovih Ni-Ti instrumenata (HyFlex) potencijalno nudi najperspektivniju metodu proizvodnje rotirajućih instrumenata [27]. Ovi instrumenti nemaju memoriju oblika koju imaju tradicionalni Ni-Ti instrumenti, a poseban termomehanički postupak značajno povećava njihovu fleksibilnost [28]. Rezultati istraživanja u ovoj studiji pokazuju najmanju kontaminiranost Hyflex sistema metalnim opiljcima. Mala zastupljenost ove kontaminacije se može objasniti njihovom specifičnom termičkom obradom koja smanjuje nepravilnosti na njihovoj površini. Toplotni tretman osim promene mikrostrukture (povećana fleksibilnost), dovodi do pojave čistije i pravilnije površine ovih instrumenata [28, 29, 29].

Prateći rezultate ove studije, zapaža se i značajno manja zastupljenost defekata u grupi BioRaCe. Ovaj nalaz je u saglasnosti sa rezultatima istraživanja o značajnom smanjenjenju površinskih nepravilnosti elektropoliranih instrumenata [3]. Elektropoliranjem se tokom procesa proizvodnje stvara homogeni oksidni sloj koji smanjuje pojavu površinskih defekata i povećava otpornost na koroziju i lom [27]. Elekropolirana površina instrumenata je vidljivo sjajnija u odnosu na netretiranu površinu [26]. Uvođenjem struje kroz rastvor dolazi do formiranja tankog pasivnog sloja i otapanja površine u elektrolit, što takođe dovodi do selektivnog uklanjanja površinskih defekata [4].

U svrhu poboljšanja mikrostrukture radne površine Ni-Ti instrumenata i poboljšavanja mehaničkih osobina, fleksibilnosti, otpornosti na zamor, odnosno sečivne efikasnosti, proizvođači poslednjih godina primenjuju različite tehnike (jonska implementacija, plazma imerzija, formiranje premaza titanijum-oksida, termalna nitridacija, termalni tretmani i kriogeni tretmani, elektropoliranje) [3, 26, 28, 30].

ZAKLJUČAK

Na osnovu rezultata ovog istraživanja može se zaključiti da su na svim ispitivanim novim instrumentima uočeni proizvodni defekti ili nečistoće (po jedan ili više). Najučestaliji tip nepravilnosti je bilo postojanje žljebova, debrisa i metalnih opiljaka na radnom delu instrumenata. Na elektropoliranoj površini BioRaCe instrumenata nije uočeno prisustvo organskog debrisa. Rezultati ove studije ukazuju na potrebu obaveznog čišćenja i sterilizacije instrumenata pre prve upotrebe ali su neophodna i dalja istraživanja u cilju dobijanja instrumenata bez defekata i nečistoća.