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# ATOMIC FORCE MICROSCOPY IN METAL FORMING AND DENTAL MATERIALS CHARACTERIZATION

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## ABSTRACT

*Atomic force microscope has been widely used for surface topography measurement in many scientific areas. In metal forming technology atomic force microscope allows analysis of various tribological phenomenon on die and produced part. Surface topography in dental restorative materials is very important since smoother tooth's surface prevents bacterial biofilm retention which is the main cause of dental pathology. This paper presents results of characterization of upset rings in friction coefficient test andresults of characterization of dental materials.*
***Keywords**: atomic force microscopy, surface roughness, dental nanomaterials, metal forming*

## 1. INTRODUCTION

Atomic force microscopy utilizes the combination of an advanced scanning probe microscope, sophisticated software, and a specific mode or technique that enables manufacturers, engineers, and researchers to precisely measure, investigate, and image the critical properties of a large variety of surfaces and samples[1](Fig. 1). It belongs to the group of devices that uses scanning techniques by probe (Scanning Probe Techniques). This group is a branch of microscopy which generates surface image by using physical tip for surface analysis [2].
Atomic force microscopy was the first technology to produce real-space images of atomic arrangements on flat surfaces and is now most commonly used to perform very precise, 3D measurements on the angstrom-to-micrometer scale [1].

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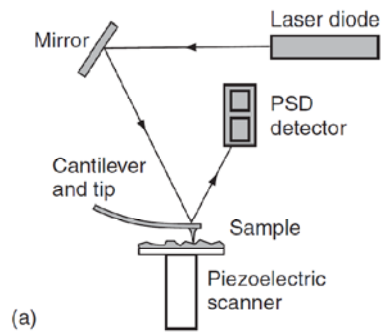



*Fig. 1 - Atomic force microscope operating principle [3]*

These properties are the main reason for AFM being helpful in researching the surface of insulators, semiconductors, polymers, composites, glass and biological samples. AFM can complete scanning in a high vacuum environment, common air and even when the tip is immersed into liquid, which is very useful for biological investigations.

## 1.1 AFM application in metal forming technology

One of the most essential problems of technology of plasticity is metal flow across die and cavity filling. These problems are especially associated with tribological state that is present on contact surface between die and workpiece.Since plastic deformation takes place in the state of a high contact stresses, which can result in a high tangential stresses too, it is very important to lower the friction coefficient. In this manner it is possible to shape complex parts and lower the amount of required operations and production cost [4-6].
The mechanisms and dynamics of the interactions of two contacting solids during relative motion, ranging from atomic to microscale, need to be understood in order to develop fundamental understanding of adhesion, friction, wear, indentation, and lubrication processes. At most solid–solid interfaces of technological relevance, contact occurs at many asperities. The importance of investigating single asperity contacts in studies of the fundamental micro/nanomechanical and micro/nanotribological properties of surfaces and interfaces has long been recognized. The emergence and proliferation of proximal probes, in particular scanning probe microscopies (the scanning tunneling microscope and the atomic force microscope) has allowed systematic investigations of interfacial problems with high resolution. These advances have led to the appearance of the new field of nanotribology, which pertains to experimental and theoretical investigations of interfacial processes on scales ranging from the atomic and molecular to the microscale, occurring during adhesion, friction, scratching, wear, indentation, and thin-film lubrication at sliding surfaces [7-16].
TheAFMcan be used to investigate how surface materials can be moved or removed on micro to nanoscales, for example, in scratching and wear (where these things are undesirable), and nanofabrication/nanomachining (where they are desirable) [8].
The key phenomenon required for understanding the subject of wear is correlation between friction coefficient and surface morphology.Friction coefficient in metal forming technology is usually determined by ring upsetting test by flat dies [4].In this study two sets of dies have been used for upsetting, ion implanted and nonimplanted dies. Ion implantation with nitrogen ions have been used, since it lowers friction coefficient [17]. Since two different values of friction coefficient are

present on the interface between the dies (implanted and nonimplanted) and sample, we investigated its influence on sample's morphology after ring upsetting test.Material flow in the samples is different in the area near the inner ring's diameter and near the outer ring's diameter and therefore the morphology in these two areas is also examined.

## 1.2 AFM application in the field of dental research

AFM could be successfully used in the characterization of either, biological dental substrates, or artificial dental restorative materials. The enamel surface can be analyzed on the submicron level using AFM; this microscopy technique obtains information about the detailed structure of the enamel tissue, and about surface changes during demineralization processes (during caries process or during non-caries mechanisms, such as erosion, abrasion, attrition) [18]. Also, the effects of toothpastes and fluoride treatment on the enamel remineralization have been examined [19-21], and the effects of acid etching and bleaching treatments, as well [22-24]. Better insight into the nanostructure of enamel can provide the detailed information that can be used for new biomimetic approaches in the synthesis of dental restorative materials. These biomimetic materials mimic natural tooth structure and appearance of dental hard tissue; it is for example: material with enamel-prism-like nanorods [25]. Dentin, as a subsurface dental hard tissue, also can be characterized by using AFM. Dentin surface texture and its organic and mineral content, can be described after tooth-preparation, or root-cavity endodontic treatment, or after treating the tooth with different mechanical and chemical procedures [26-29]. In addition to the characterization of dental biological structures, artificial dental materials can also be well described by AFM analysis. Dental materials, such as zirconium-oxide ceramic [30], [31], lithium di-silicate glass ceramic [32], alumina ceramic [33], resin-based composites (RBCs) [34], [35], flowable composites [36] and nanocomposites[37] has been already examined in several studies. Researchers explore surface modification techniques of dental titanium implants, as well, in order to provide better biological response and tissue adhesion on the implant surface. In these studies, AFM serves as an excellent methodology choice for the implant surface characterization on the submicron level [38].
In this study we have determined surface roughness of polished contemporary dental resin-based composites, using atomic force microscopy analysis. These materials are tooth-colored, highly esthetic and the most commonly used biomaterials in contemporary dental practice [39]. RBCs consist of three different phases: organic resin matrix, inorganic filler particles and silane- the filler-resin interface [40]. They can be classified according to their filler composition in three main groups: microhybrid, microfilled and nanofilled materials. Among these groups, there are significant differences in mechanical, optical and esthetic material properties, and also the differences in the polish-ability and polish retention and wear behavior [41]. We investigated the surface characteristics of these materials after finishing and polishing by standard dental polishing procedures.
The aim of this study was to determine surface roughness of polished contemporary dental RBCs using atomic force microscopy analysis.

## 2. AFM APPLICATION IN METAL FORMING TECHNOLOGY

### 2.1 Materials and methods

Fig. 2 shows contact friction coefficient determination by ring upsetting method.Method consists of establishing the dependence between deformation of inner ring's diameter and ring's height. This dependence is taken into etalon chart and compared with the existing within the chart.
Ring upsetting has been performed incrementally, with a height deformation around 10%. After each upsetting stage ring's dimensions were measured. Incremental upsetting has been carried out until total deformation of the ring's height has reach around 70%.
Once the ring upsetting has been completed, the deformation of the ring's inner diameter and the deformation of the ring's height have been calculated for each upsetting increment. By connecting all the pairs of height and inner diameter deformation,the curve was defined and inserted into etalon diagram.
In order to find the friction factor for the completed upsetting process, it is necessary to compare the curve obtained from the experiment with the existing ones from the etalon diagram.
Ring upsetting has been carried out with two different pairs of dies.
One pair of dies has been grinded, polished and ion implanted with $2 \cdot 10^{17}$ $N^+$ 50 keV, while another pair of dies has not been ion implanted.

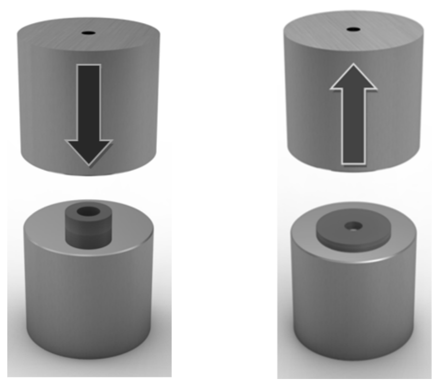


*Fig.2 - Ring upsetting method*

Dies were made of X210Cr12 cold work tool steel (Č.4150) with dimensions ø50×45 mm. Rings were made of Ck15 unalloyed carbon steel (Č.1221) with initial dimensions $D_2$:$D_1$:h=18:9:6 mm. Hardness of the dies was 58+2 HRC, while hardness of the ring upset with nonimplanted dies was 167 HV-10 and hardness of the ring upset with implanted dies was 161 HV-10. Upsetting was done without contact surface lubrication.
Relatively simple ring shape has asymmetric material flow during upsetting process. Simulation software Simufact.forming has been used for material flow phenomena analysis. Two distinctive zones (A and B) of material flow inside the rings have been noticed after upsetting (Fig. 3). Zone A has almost zero value of horizontal material flow. Contrary, B zone is distinguished by maximal value of horizontal flow over the tool surface.

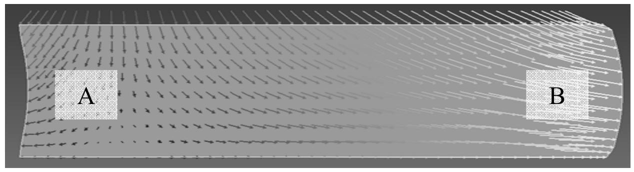



*Fig. 1 - Simufact.forming results of ring upsetting with implanted dies*

Purpose of sample's topography measurement prior and after ring upsetting was to determine the influence of die's surface ion implantation on friction and dimensional accuracy, i. e. workpiece surface quality in metal forming processes. By comparing the roughnesses between corresponding rings, the influence of die's surface ion implantation on ring's quality and accuracy can be established. Topography of the ring was measured with VEECO "di CP II" atomic force microscope.

## 2.2 Results and discussion

Fig. 4 shows comparison of friction factors obtained from a ring upsetting experiment while tab. 1 shows friction factors and friction coefficients values.
To evaluate the effect of die surface ion implantation on ring's surface roughness during upsetting, ring roughness has to be determined before upsetting was carried out. Fig. 5 displays topography of the ring that hasn't been upset with dies. The average $R_a$ values of the rings before upsetting were around 130 nm.

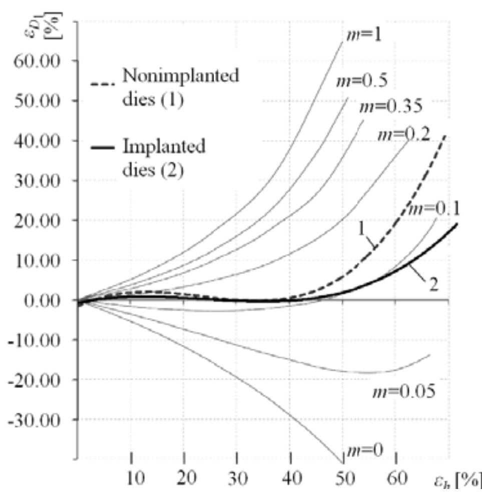



*Fig. 2 - Comparison of friction factors for rings upset with implanted (2) and nonimplanted dies (1)*

*Table 1 - Friction factors and friction coefficients for rings upset with implanted and nonimplanted dies*

| Dies | Friction factor (m) | Friction coefficient (μ) |
|---|---|---|
| Nonimplanted | 0.15 | 0.087 |
| Implanted | 0.11 | 0.064 |

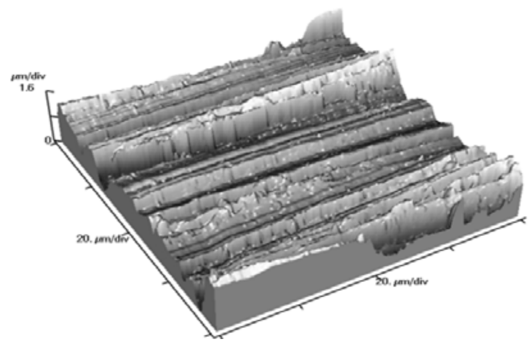



*Fig. 3 - Topography of the ring's surface before upsetting*

Fig. 6 shows measuring points where surface roughness was measured using atomic force microscopy. The distance between measuring points on the rings is approximately 1 mm.

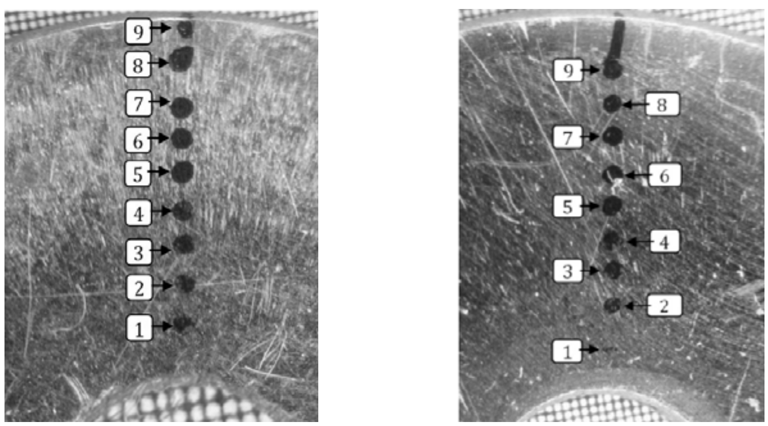



*Fig. 4 - Measuring points on the ring upset with*
*a) nonimplanted dies, b) implanted dies*

Fig. 7 shows topography of the ring upset with nonimplanted dies at measuring point X7 (see Fig. 6), while Fig. 8 shows topography of the ring upset with implanted dies also at measuring point X7. The biggest difference in terms of $R_a$ values between the upset rings is in measuring point X7 (Fig. 9).

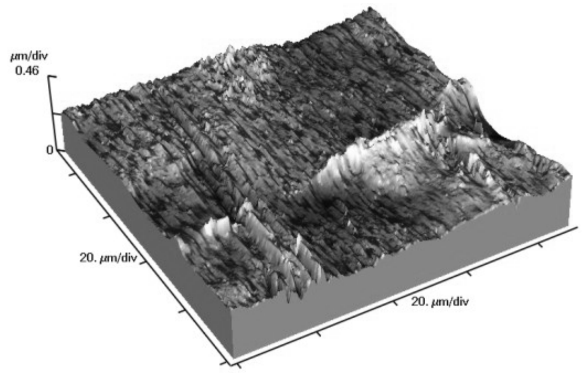



*Fig. 5 - Topography of the ring upset with nonimplanted dies measured at point X7*

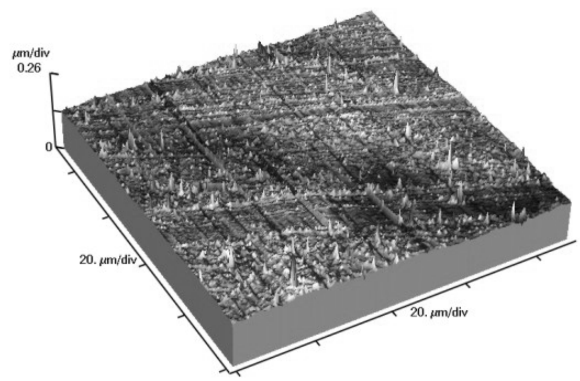



*Fig. 6 - Topography of the ring upset with ion implanted dies measured at point X7*

Fig. 9 shows roughness comparison between the rings upset with nonimplanted and ion implanted dies.

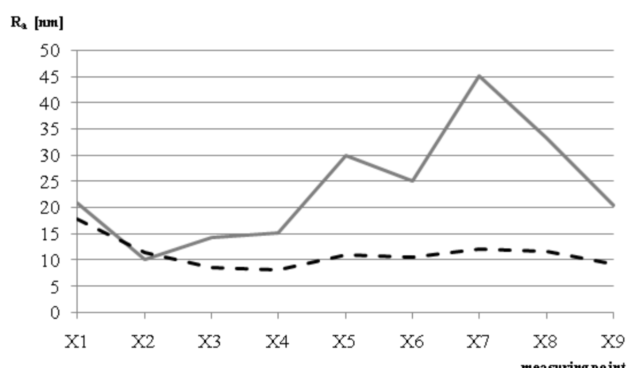



*Fig. 7 - Roughness comparison between the rings upset with nonimplanted and ion implanted dies*

It is evident from the diagram (Fig. 4) that ion implantation has the influence on friction coefficient in ring upsetting process, since friction coefficient is 1.36 times lower in case of upsetting with ion implanted dies.
It is obvious from Fig. 7 and Fig. 8 that rings upset with ion implanted dies has smoother surface compared to ring upset with nonimplanted dies.
Diagram on Fig. 9 shows that ring upset with ion implanted dies has roughness in narrower range compared to ring upset with nonimplanted dies and that at X7 measuring point, the difference in $R_a$ values between the rings can be almost four times.Also, $R_a$ in zone B in ring upset with nonimplanted dies (measuring points X5-X9) is higher than in zone A (X1-X4) because of the horizontal material flow present in zone B during ring upsetting.

## 3. ROUGHNESS DETERMINATION OF POLISHED CONTEMPORARY DENTAL RESIN BASED COMPOSITES

### 3.1 Materials and methods

Three representative dental resin-based composites were tested in the study: nanofilled (Filtek Ultimate Translucent, 3M ESPE, St. Paul, MN, USA), microfilled (GC Gradia Direct Anterior, GC Dental Products Corporation, Tokyo, Japan) and microhybrid (Filtek Z250, 3M ESPE, St. Paul, MN, USA).
One specimen of each material was made by using cylindrical plastic molds (4 mm diameter x 2 mm depth). Plastic molds were placed on the glass microscope slide, filled with material and covered with a polyester strip and a glass slide, taking care to obtain a flat surface without any defects and entrapped air. Material was then polymerized for 40 sec. with a SmartLite® IQTM 2 LED unit (Dentsply Caulk). After removing glass plate and polyester strip from the top of the samples, they were polished with multi-step polishing system- Super Snap (Shofu, Inc. Kyoto, Japan).
During the polishing procedure, each abrasive disk was used only once for each material, in the dry condition, for 1 minute, using handpiece rotating 10000 revolutions per minute (recommended speed by manufacturer). Four different abrasive disks were used during polishing procedure: black (coarse), violet (medium), green (fine) and red (extra-fine). One single operator did all of the polishing treatments, trying to simulate clinical finishing and polishing procedure. Two mutually perpendicular grinding directions were used during polishing procedure (Fig. 10).
Immediately after the polishing treatment, topography of each specimen was examined by Veeco di CP-II Atomic Force Microscope. Specimen's surface has been scanned at points which lie at half distance between specimen's center and perimeter in contact mode with CONT20A-CP tips. 1 Hz scan rate and 256 × 256 resolution were used to obtain topography on a 90 μm × 90 μm scanning area. Before the scanning, specimen's surface has been blown through with cold air by hairdryer. Cleaning specimen's surface with alcohol created damaged surface.
Once AFM images were obtained, surface roughness analysis was carried out on each AFM image, beginning by identifying the grinding tracks. After that, three analyzing lines along and three analyzing lines perpendicular to the grinding tracks were set on each AFM image, so that the roughness parameters in each line can be calculated. The purpose of setting the analyzing lines in this way was to compare roughness parameters in line and perpendicular to grinding tracks and to discover the influence that abrasive discs had on each material.

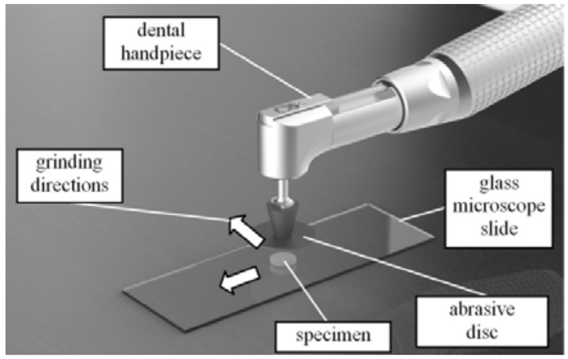
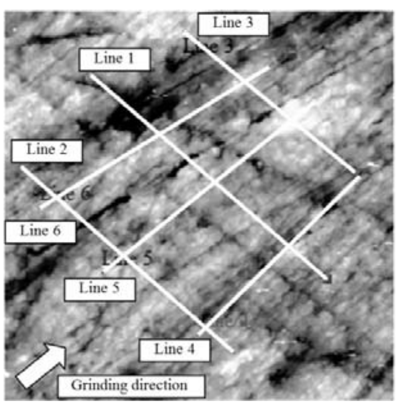



*Fig. 8. Grinding setup and grinding directions*

Measured topography data were processed by Image Processing and Data Analysis v2.1.15 software. Following parameters were compared among specimens: average roughness ($R_a$) and maximum peak-to-valley distance ($R_{p-v}$).

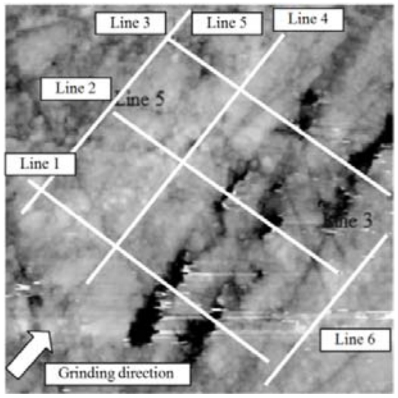
## 3.2 Results and discussion

Figs.11, 12 and 13 displays AFM images obtained from the surface of each specimen and tables which contain values of ($R_a$) and ($R_{p-v}$) roughness parameters from each analyzing line. Fig.14 displays comparison of roughness parameters between all specimens, along and perpendicular to grinding tracks.



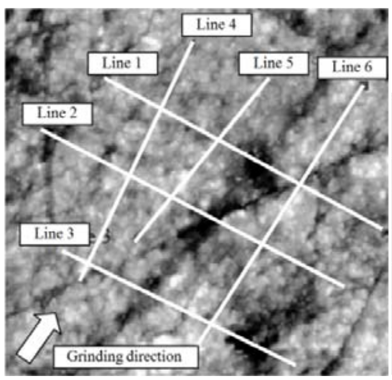

| Measuring location | $R_a$ [nm] | $R_{p-v}$ [nm] |
|---|---|---|
| Line 1 | 57.91 | 380.9 |
| Line 2 | 32.04 | 239.0 |
| Line 3 | 36.64 | 193.4 |
| Line 4 | 26.16 | 214.7 |
| Line 5 | 29.82 | 193.3 |
| Line 6 | 29.28 | 165.6 |

*Fig. 9 - a) AFM image and analyzing lines for Filtek Ultimate Translucent (nanofilled)*
*b) $R_a$ and $R_{p-v}$ parameters on analyzing lines for Filtek Ultimate Translucent*




| Measuring location | $R_a$ [nm] | $R_{p-v}$ [nm] |
|---|---|---|
| Line 1 | 87.62 | 786.5 |
| Line 2 | 94.66 | 745.4 |
| Line 3 | 85.68 | 774.7 |
| Line 4 | 37.24 | 194.1 |
| Line 5 | 48.92 | 333.3 |
| Line 6 | 49.16 | 401.7 |

*Fig. 10 - a) AFM image and analyzing lines for GC Gradia Direct Anterior (microfilled)*
*b) $R_a$ and $R_{p-v}$ parameters on analyzing lines for GC Gradia Direct Anterior*




| Measuring location | $R_a$ [nm] | $R_{p-v}$ [nm] |
|---|---|---|
| Line 1 | 43.27 | 375.8 |
| Line 2 | 37.52 | 351.4 |
| Line 3 | 37.81 | 284.5 |
| Line 4 | 21.25 | 154.2 |
| Line 5 | 31.82 | 174.7 |
| Line 6 | 29.44 | 162.9 |

*Fig. 11 - a) AFM image and analyzing lines for Filtek Z250 - (microhybrid)*
*b) $R_a$ and $R_{p-v}$ parameters on analyzing lines for Filtek Z250*

Based on the values of surface roughness parameters it can be concluded that Filtek Ultimate Translucent had the lowest values of ($R_a$) and ($R_{p-v}$) compared to GC Gradia Direct Anterior and Filtek Z250 (Fig. 14), which confirms the existence of advanced material features due to the nanoparticles filler composition. Also, when comparing the same roughness parameters along and perpendicular to grinding tracks, Filtek Ultimate Translucent showed the best behavior in terms of surface uniformity after grinding by abrasive discs - there were the smallest differences in terms of roughness values in the both analyzing directions. In all cases, measuring roughness along grinding tracks showed lower values than perpendicular to grinding tracks.

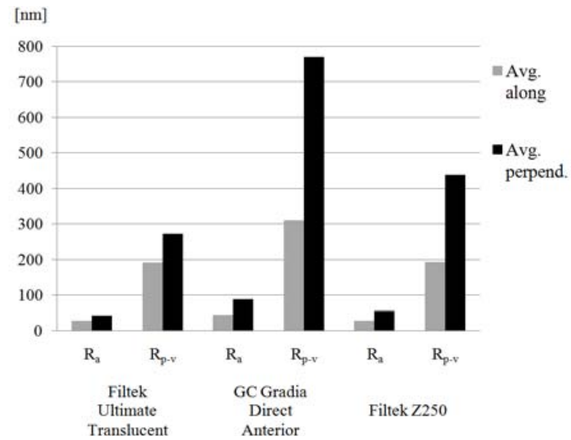



*Fig. 12 - Comparison of roughness parameters among dental resin-based nanocomposites along and perpendicular to grinding tracks*

## 4. CONCLUSIONS

Based on the results presented in this paper, it can be concluded that ion implantation can reduce the friction coefficient and improve surface roughness and quality at bulk forming process. AFM application is essential for researching surface nanomorphology in bulk forming processes. Results obtained in this paper contribute to the development of ultraprecision engineering.

Based on obtained values of surface roughness parameters it can be concluded that nanofilled resin composite had the lowest surface roughness among the three tested groups of resin-based composites and the highest surface uniformity after dental finishing and polishing procedure. Smoother material surface prevents bacterial biofilm retention which is the main cause of dental and periodontal pathology. Any improvement of the material properties is allowing better therapeutic possibilities.

This paper presented representative results that reflect the broad range of AFM applications in many different research areas. Possible applications of this microscopy technique are great and they allow interdisciplinary research and cooperation in various research fields.

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# MIKROSKOPIJA ATOMSKIM SILAMA U OBRADI METALA DEFORMISANJEM I KARAKTERIZACIJA STOMATOLOŠKIH BIOMATERIJALA

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## REZIME

*Atomski mikroskop se u velikoj meri koristi za određivanje topografije površina u mnogim naučnim oblastima. U prezentovanom radu prikazane su mogućnosti primene atomskog mikroskopa u dvema različitim oblastima - u obradi metala deformisanjem i u karakterizaciji stomatoloških materijala. U obradi metala deformisanjem, atomski mikroskop omogućuje analizu različitih triboloških fenomena na alatima i proizvedenim delovima. Kada je reč o karakterizaciji stomatoloških materijala, atomski mikroskop određuje kvalitet površinske topografije stomatoloških restaurativnih materijala, koji je od velike kliničke važnosti, jer glatka površina zubnog ispuna sprečava retenciju biofilma – glavnog uzroka zubne patologije i patologije potpornog aparata zuba. Ovaj rad predstavlja rezultate karakterizacije sabijenih prstenova u testu određivanja trenja i rezultate karakterizacije stomatoloških kompozitnih materijala na bazi smola.*
*Ključnereči: atomski mikroskop, površinska hrapavost, stomatološki kompozitni materijali na bazi smola, obrada metala deformisanjem*