

Determination of surface roughness and topography of dental resin-based nanocomposites using AFM analysis

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ABSTRACT

The aim of this study was to determine surface roughness and topography of polished dental resin-based nanocomposites.

Four representative dental resin-based nanocomposites were tested in the study: two nanohybrids (Filtek Z550 and Tetric EvoCeram) and two nanofilled (Filtek Ultimate Body and Filtek Ultimate Translucent); and two reference materials: one microfilled (Gradia Direct) and one microhybrid (Filtek Z250). Polymerized cylindrical specimens (4 mm x 2 mm) were polished with multi-step polishing system- Super Snap. Immediately after the polishing, topography of each specimen was examined by Veeco di CP-II Atomic Force Microscope. Specimen's surface has been scanned in 6 points in contact mode with CONT20A-CP tips. 1 Hz scan rate and 256 x 256 resolution were used to obtain topography on a 90 µm x 90 µm scanning area. Measured topography data were processed by Image Processing and Data Analysis v2.1.15 software. Following parameters were compared among specimens: average roughness and maximum peak-to-valley distance.

All of the tested materials had similar average surface roughness after finishing and polishing procedure. The lowest values occurred in the material Filtek Ultimate Body, and the highest in the Filtek Z550. When interpreting maximum peak-to-valley distance the larger differences in values (up to 100%) occurred in Filtek Z550, Filtek Z250 and Filtek Ultimate Body, which is a result of the deep polishing channels and tracks. Type, size, distribution of fillers and filler loading in tested materials, didn't influence average roughness values, but had an impact on maximum peak-to-valley distance values.

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KEY WORDS: dental polishing and finishing, resin-based composite, nanocomposite, nanoparticles, atomic force microscopy, surface roughness, topography

INTRODUCTION

Nanotechnology is the science and engineering involved in the design, synthesis, characterization and application of materials and devices whose smallest functional organization in, at least, one dimension is on the nanometer scale (one-billionth of a meter, 10^{-9} m) [1]. Something that we call nanomaterial or nanodevice has the smallest dimension about 100 nm or less, i.e. maximum 1×10^{-7} m. Nanotechnology is a multidisciplinary field of scientific research, which can be used in the numerous application areas [2]. Potential benefits of using nanomaterials and nanorobots in medical and dental applications are one of the main topics when talking about nanoadvances [3]. In the field of dentistry, nanotechnological improvements may lead to advanced pre-

ventive, diagnostic and therapy procedures [4, 5, 6]. Currently, nanoproducts that have the widest application in routine dental clinical practice are resin-based nanocomposites. Dental resin-based composites (RBC) are tooth-colored restorative materials that consist of organic resin matrix, inorganic filler particles, silane coupling agents and initiators and activators for the photo-polymerization [7]. Overall characteristics of the material may be improved by the research and work on improving the individual components of RBCs [8]. Fillers in composites have multiple roles: to reduce polymerization shrinkage, the coefficient of thermal expansion and water sorption and solubility; to mechanically reinforce the material; to improve optical and aesthetic characteristics of the material; to enable better initial polishing and polish retention, and to reduce wear during the masticatory forces [7, 9-13]. Formulation of filler particles, have been passed from macro-, micro-, down to the nano-particles [14]. Microhybrid composites, so-called universal restorative composites, are composed of filler particles of different sizes (15-20 µm and 0.01-0.05 µm) and have good mechanical properties for use in the lateral occlusal region, but relatively poor aesthetic qualities, to be used in the esthetic zone [15]. Microfilled

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composites have been developed in order to obtain high-quality aesthetic materials that meet the needs of restorative dentistry in the esthetic zone. Microfilled composites have average particle size in range of 0.01-0.05 μm . Due to the relative poor mechanical strength, these materials are indicated for use in low-stress oral regions [15]. Trying to create a material that meets both of these properties, the mechanical resistance and the aesthetic and polishing qualities, nanofillers have been developed [12]. In order to produce particles smaller than 100 nm, the mode of production of particles for RBCs had to be changed from the “top-down” milling and grinding procedure to the “bottom-up” manufacturing approach (direct molecular assembly) [12, 16]. Current dental resin-based nanocomposites can be divided into two main groups: nanohybrids and nanofilled composites (nanofills) [16]. Nanohybrids consists of particles of various sizes, particles in micrometric and in nanometric range [7]. Nanofills consist of particles of nearly uniform size, all in nanometric dimensions, with the ability to create nanoclusters as secondarily formed fillers [7, 12]. Nanocomposites have excellent mechanical properties, similar as microhybrids, which make them able to withstand high occlusal forces, in lateral masticatory regions. On the other hand, they have great polish ability and polish re-

tention, superior optical and aesthetic characteristics [12]. Surface quality of resin-based composite is a very important characteristic of the final restoration. Smooth surface of the restoration is necessary to obtain clinical durability and good aesthetic appearance, and to prevent discoloration and staining. Also, dental plaque, as a main cause of periodontal diseases and secondary caries, is less retained on the smooth surface [17]. Polishing treatments can improve the wear behavior of the material, as well [18, 19]. Finishing procedure is a necessary clinical step to establish a proper reconstruction of dental crowns and to restore anatomical and morphological form of the tooth. Another reason for this procedure is to remove the resin-rich surface layer, which remains after polymerization and removal of the matrix from the material [20]. Polishing procedure makes the surface smoother and removes surface damages like grooves, lines and furrows created during finishing phase [18]. Aim of this study was to determine surface roughness and topography of contemporary RBCs using atomic force microscopy analysis. The two main goals were: (a) analysis of the impact of particle size and loading on the surface roughness and topography after finishing and polishing procedure, and (b) the impact of the multi-step finishing and polishing procedure, itself, on the surface topography of RBCs.

TABLE 1. Details of the materials tested in the study*

| Material | Manufacturer | Classification | Lot no. | Shade | Matrix | Fillers | Filler loading |
|-----------------------------|--|---------------------------------|---------|-------------|--|---|---|
| Filtek Z550 | 3M ESPE, St. Paul, MN, USA | Nanohybrid | N340139 | A2 | Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA | Surface-modified zirconia/silica fillers 3000 nm (3 μm or less), non-agglomerated/non-aggregated surface-modified silica particles 20 nm | 82 wt % 68 vol % |
| Tetric EvoCeram | Ivoclar Vivadent, Schaan, Liechtenstein | Nanohybrid | P80726 | A2 | Bis-GMA, UDMA | Barium glass, ytterbium trifluoride, mixed oxide and prepolymer; 40-3000 nm, 550 nm | 82-83 wt % 82.5 vol% |
| Filtek Ultimate Body | 3M ESPE, St. Paul, MN, USA | Nanofilled | N349776 | A2 | Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA | non-agglomerated/non-aggregated 20 nm silica filler; non-agglomerated/non-aggregated 4-11 nm zirconia filler; and aggregated zirconia/silica cluster filler (average cluster particle size – 0.6-10 μm) | 78.5 wt%, 63.3 vol% |
| Filtek Ultimate Translucent | 3M ESPE, St. Paul, MN, USA | Nanofilled | N225533 | Clear shade | Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA | non-agglomerated/non-aggregated 20 nm silica filler; non-agglomerated/non-aggregated 4-11 nm zirconia filler; and aggregated zirconia/silica cluster filler (average cluster particle size – 0.6-20 μm) | 72.5 wt%, 55.6 vol% |
| GC Gradia Direct Anterior | GC Dental Products Corporation, Tokyo, Japan | Microfilled (Micro-fine hybrid) | 1106011 | A2 | UDMA, dimethacrylate co-monomers, -II- | Silica, 850 nm (0.85 μm) and prepolymerized filler | 73 wt% 64-65 vol% (silica- 38 wt %, 22 vol %; prepolymerized filler- 35 wt%, 42 vol%) |
| Filtek Z250 | 3M ESPE, St. Paul, MN, USA | Microhybrid, St Paul, MN, USA | N367949 | A2 | Bis-GMA, UDMA, Bis-EMA, TEGMA | Zirconia, silica 10 – 3500 nm (0.01-3.5 μm) | 75-85 wt% 60 vol% |

Bis-GMA- bisfenol A diglicidil ether dimethacrylate; Bis-EMA- bisfenol A polyethylene glycol diether dimethacrylate; UDMA- urethane dimethacrylate; TEGMA- triethyleneglycol dimethacrylate; PEGDMA- polyethylene glycol dimethacrylate

*Data obtained from the manufacturers

MATERIALS AND METHODS

Materials

Four representative dental resin-based nanocomposites were tested in the study: two nanohybrids (Filtek Z550 and Tetric EvoCeram) and two nanofilled (Filtek Ultimate Body and Filtek Ultimate Translucent); and two reference materials: one microfilled (Gradia Direct) and one microhybrid (Filtek Z250). Detailed information about material used in the study are shown in the Table 1.

Procedure for preparing the specimens

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 seconds 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 10 000 revolutions per minute (recommended speed by manufacturer). 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 (Figure 1). Detailed information about the polishing system used in the study are shown in Table 2. 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 in 6 points (two points at specimen's center, two points at specimen's perimeter and two points at half distance between specimen's center and perimeter (Figure 2) 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. 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}).

RESULTS

The results obtained by this study are presented in Table 3 displaying measured values of (R_a) and (R_{p-v}) roughness parameters of specimens used, and in Figure 3 and 4 where these values are graphically presented. Figures 5 to 10 present 3D images of surface topography on each specimen obtained by AFM. Each figure contains six 3D AFM images that are obtained on each measuring point on specimen (two points

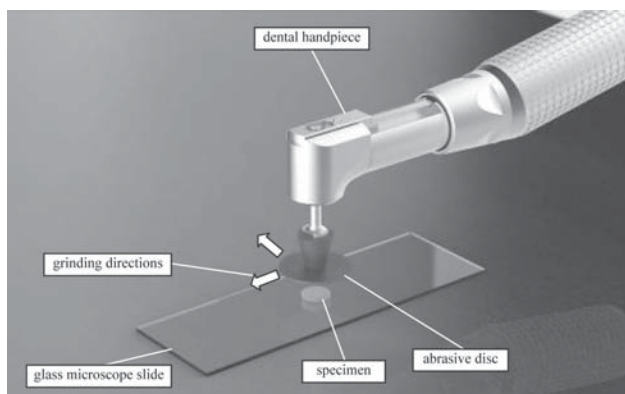


FIGURE 1. Grinding setup and grinding directions



FIGURE 2. AFM measurement points on specimen's surface. (1 - specimen's center, 2 - half-distance between specimen's center and circumference, 3 - specimen's circumference)

TABLE 2. Details about polishing system used in the study

| Name | Manufacturer | Usage | Handpiece speed |
|----------------------------|--------------------------|---|-----------------|
| Super Snap* (multi - step) | Shofu, inc. Kyoto, Japan | 1 minute polishing, dry condition, black (coarse) abrasive disk | 10 000 rpm* |
| | | 1 minute polishing, dry condition, violet (medium) abrasive disk | 10 000 rpm* |
| | | 1 minute polishing, dry condition, green (fine) abrasive disk | 10 000 rpm* |
| | | 1 minute polishing, dry condition, red (extra-fine) abrasive disk | 10 000 rpm* |

*recommended rpm by manufacturer

TABLE 3. Values of (R_a) and (R_{p-v}) surface parameters on specimens used in this study

| Measur. Point | Filtek Z550 | | Tetric EvoCeram | | Filtek Ultimate Body | | Filtek Ultimate Translucent | | GC Gradia Direct Anterior | | Filtek Z250 | |
|---------------|-------------|----------------|-----------------|----------------|----------------------|----------------|-----------------------------|----------------|---------------------------|----------------|-------------|----------------|
| | R_a [nm] | R_{p-v} [nm] | R_a [nm] | R_{p-v} [nm] | R_a [nm] | R_{p-v} [nm] | R_a [nm] | R_{p-v} [nm] | R_a [nm] | R_{p-v} [nm] | R_a [nm] | R_{p-v} [nm] |
| Point 1 | 34.97 | 756.3 | 27.25 | 474.5 | 20.79 | 282.6 | 42.68 | 661.6 | 38.72 | 686.7 | 30.01 | 635.6 |
| Point 2 | 51.94 | 1219 | 34.26 | 552 | 40.54 | 720.9 | 38.39 | 651.8 | 58.00 | 934 | 40.09 | 807.3 |
| Point 3 | 60.82 | 1270 | 44.33 | 925.8 | 46.29 | 917.5 | 42.78 | 896 | 46.98 | 1390 | 31.25 | 732.5 |
| Point 4 | 82.40 | 1670 | 42.63 | 681.1 | 31.68 | 553.1 | 38.91 | 630.1 | 72.93 | 1134 | 36.98 | 1139 |
| Point 5 | 45.97 | 704.4 | 38.15 | 533.5 | 33.75 | 516.9 | 43.00 | 992 | 47.73 | 909.3 | 35.22 | 878.4 |
| Point 6 | 42.62 | 1308 | 36.42 | 427.8 | 18.90 | 435 | 32.83 | 470.6 | 46.41 | 884.8 | 50.26 | 741.7 |

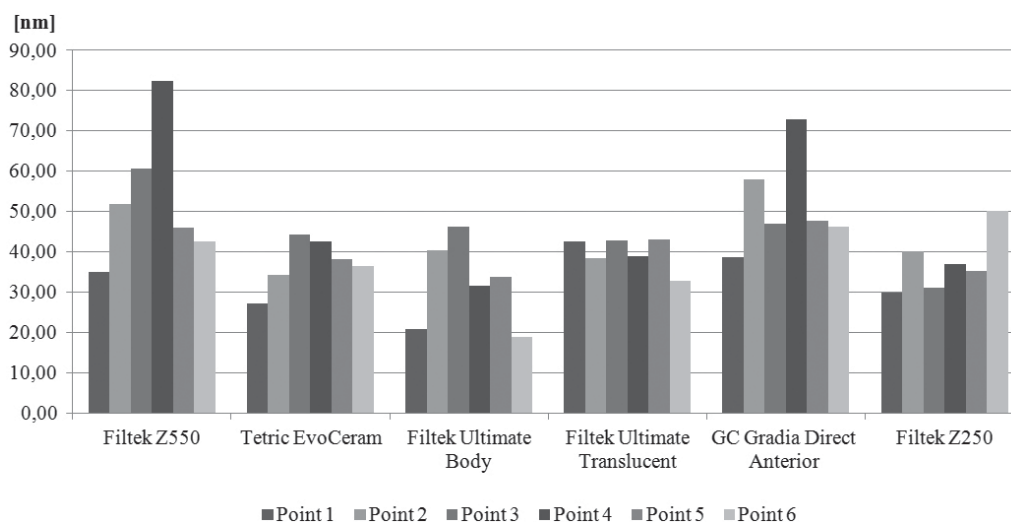


FIGURE 3. Comparison of average roughness (R_a) among specimens after polishing

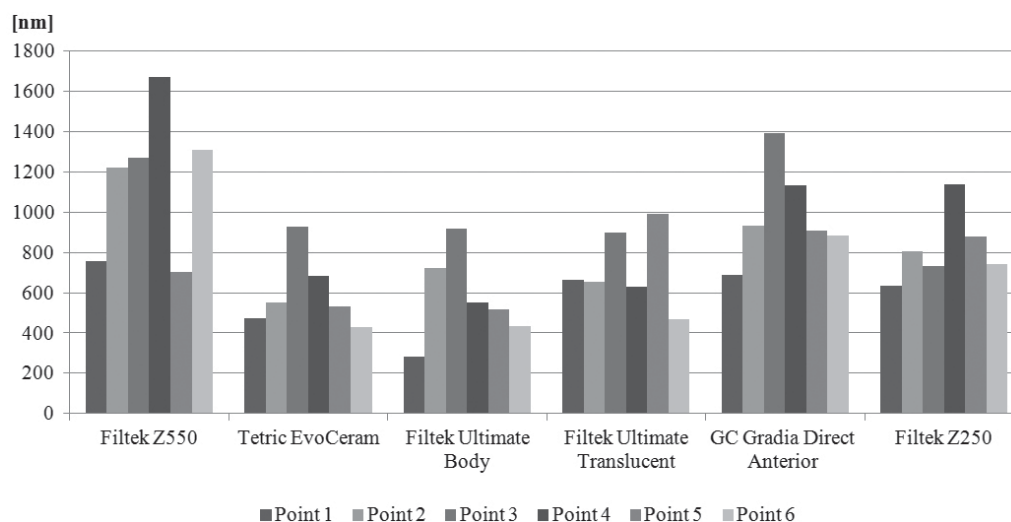


FIGURE 4. Comparison of peak-to-valley (R_{p-v}) distance among specimens after polishing

on specimen's center, two points on specimen's perimeter and two points at half distance between specimen's center and perimeter). From the analysis of the results showed in Figure 3, it can be seen that although there are different materials tested in the study (nanohybrids, nanofills, microfills and microhybrids- with different filler composition, size and

volumetric loading of the material), all the materials have similar average surface roughness (R_a) after finishing and polishing procedure. The lowest (R_a) values occurred in the material Filtek Ultimate Body, and the highest in GC Gradia Direct and Filtek Z550. Filtek Ultimate Translucent and Tetric EvoCeram showed the most consistent results of all

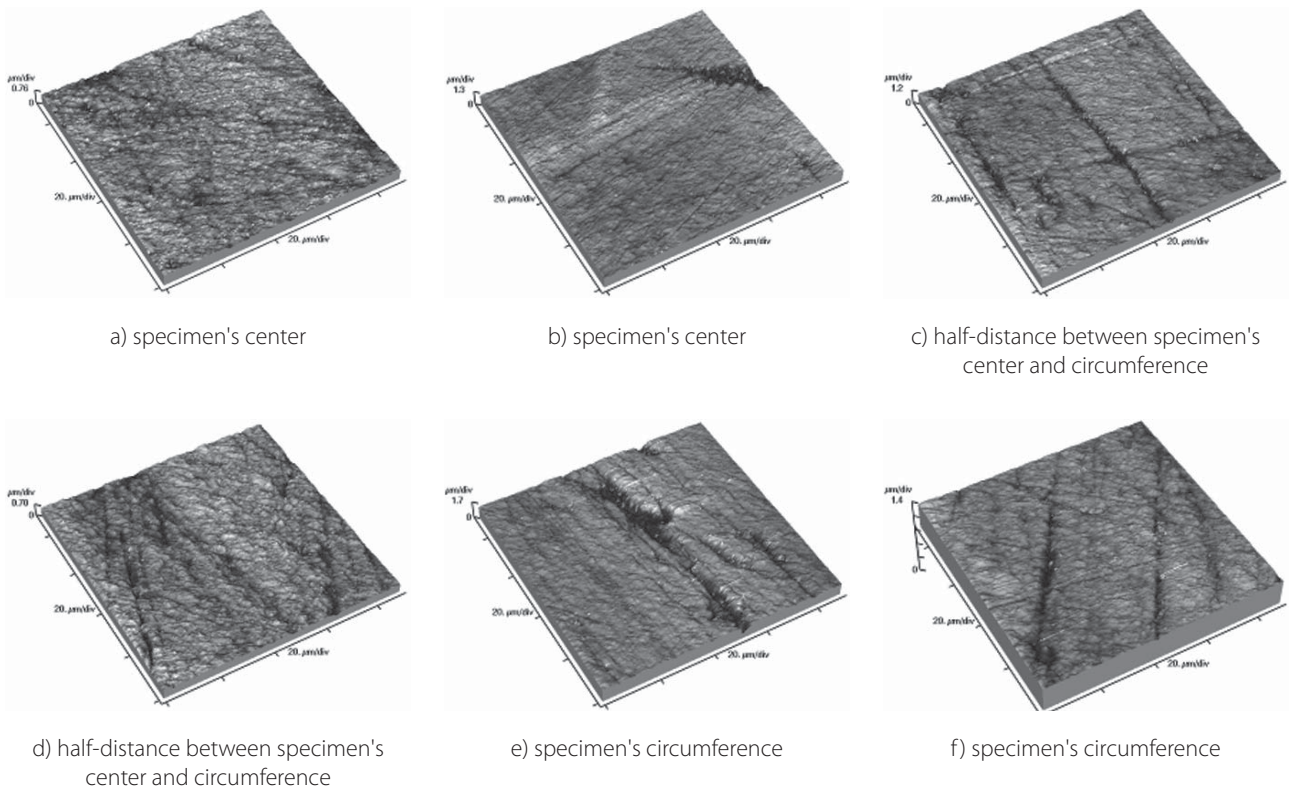


FIGURE 5. Topography of Filtek Z550 sample

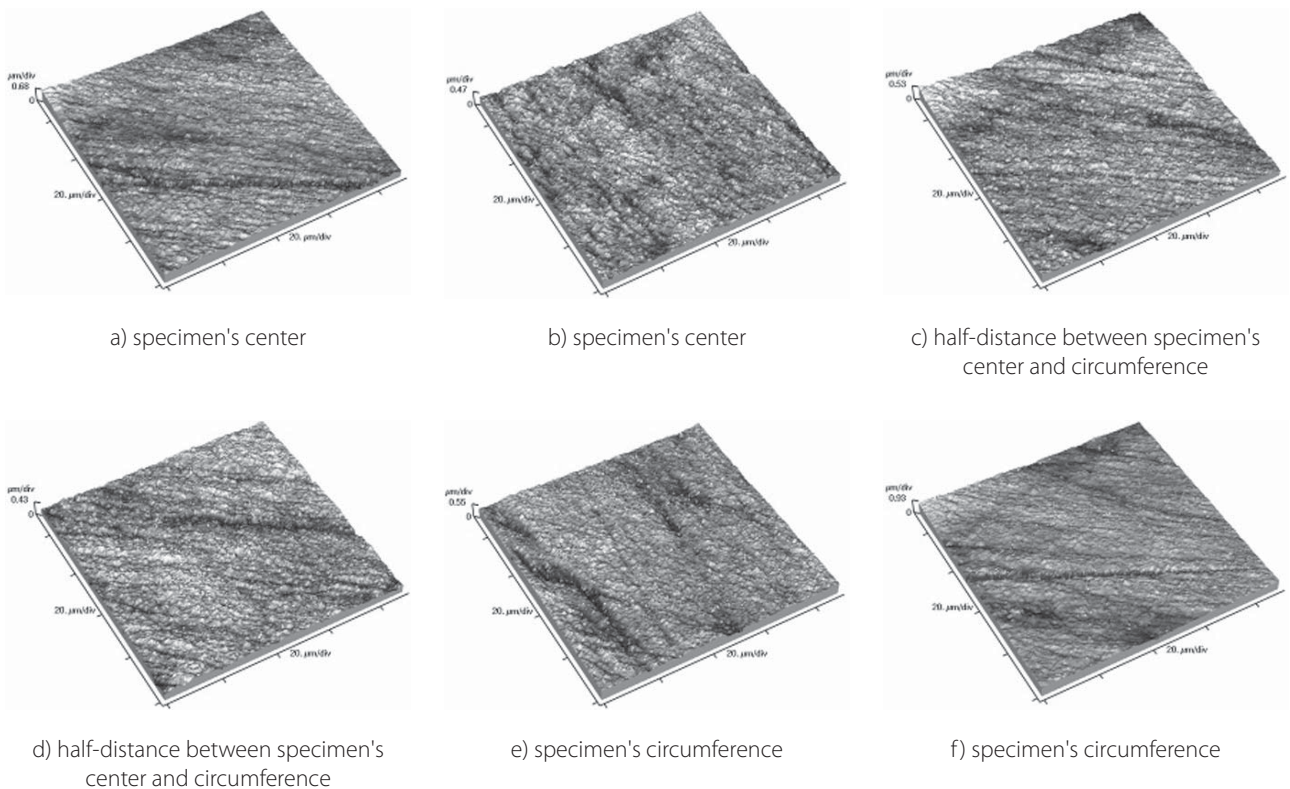


FIGURE 6. Topography of Tetric EvoCeram sample

the measurements on the sample (Figure 3), which can be confirmed by the uniform 3D AFM images, as well (Figure 8, and Figure 6). When interpreting (R_{p-v}), the larger differ-

ences in values (up to 100%) occurred in Filtek Z550, Filtek Z250 and Filtek Ultimate Body, which is the result of the deep polishing channels and tracks. During qualitative analysis of

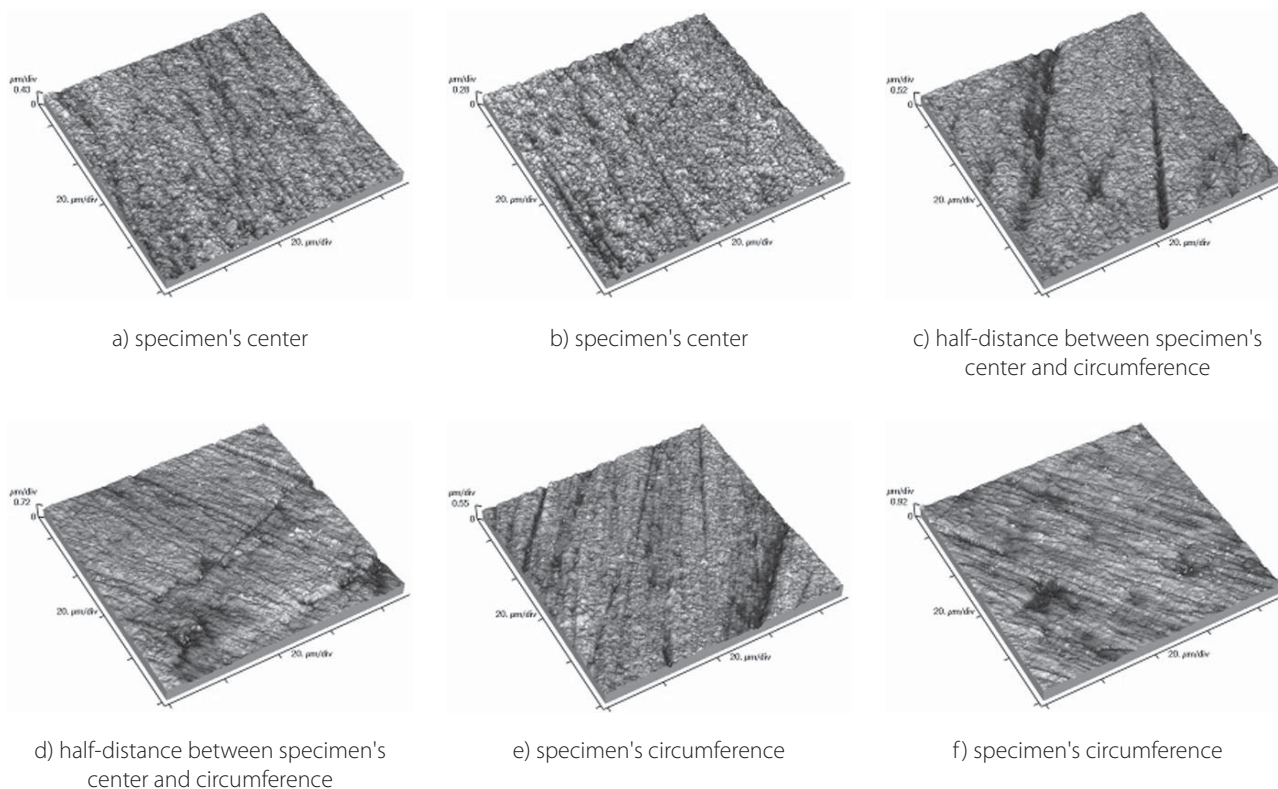


FIGURE 7. Topography of Filtek Ultimate Body sample

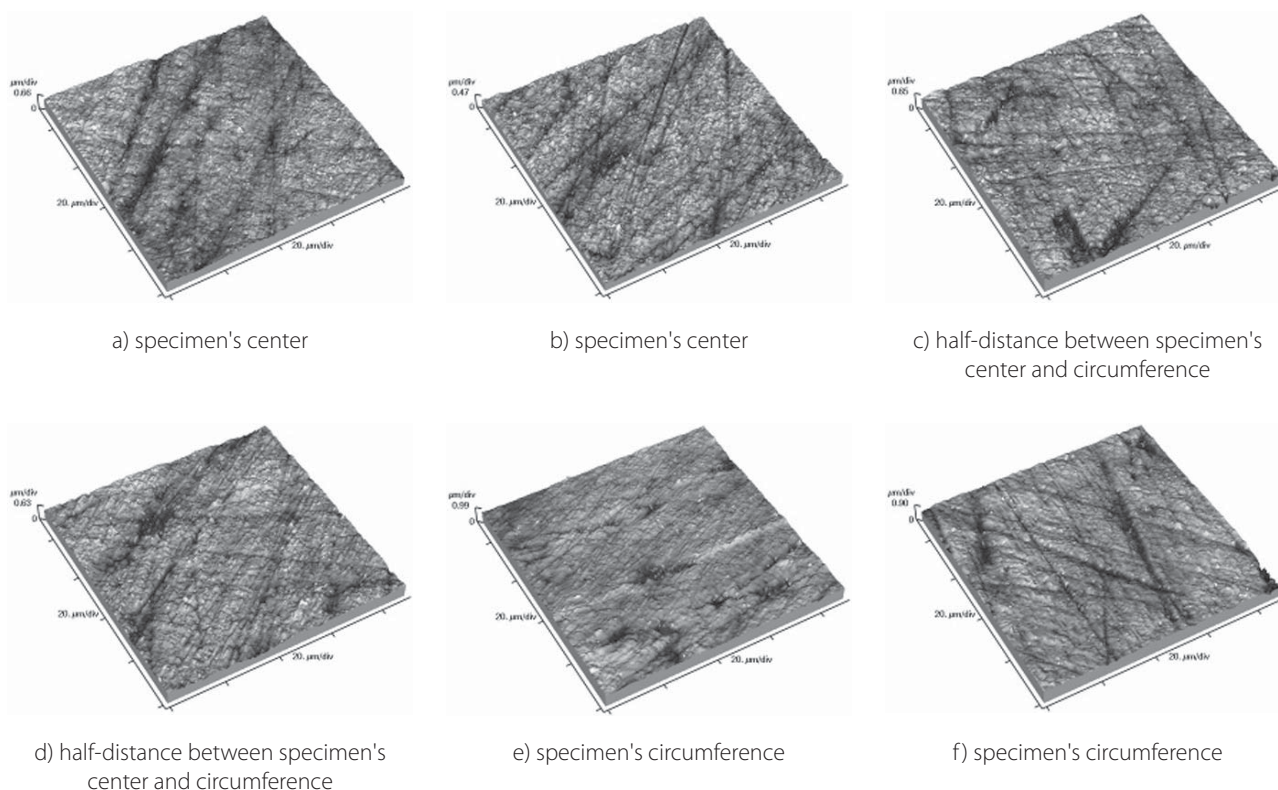


FIGURE 8. Topography of Filtek Ultimate Translucent sample

3D AFM images it can be seen that material surfaces contain various channels and polishing tracks of different size, flat areas or local damages. By analyzing images it can be noticed

that Tetric EvoCeram exhibits highly-uniform surface quality on all of the measuring points (Figure 6). Visible polishing track and local damages feature GC Gradia Direct's surface

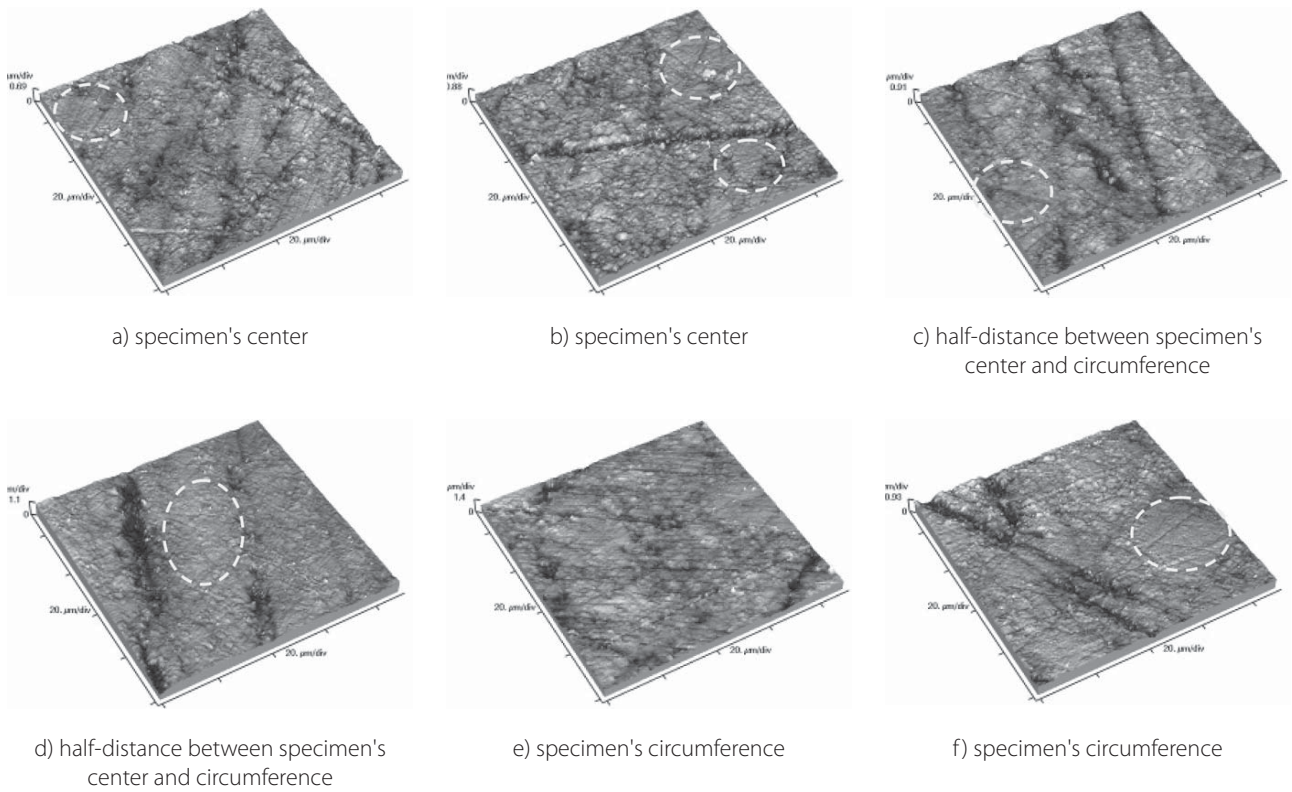


FIGURE 9. Topography of GC Gradia Direct Anterior sample with indicated flat surface regions

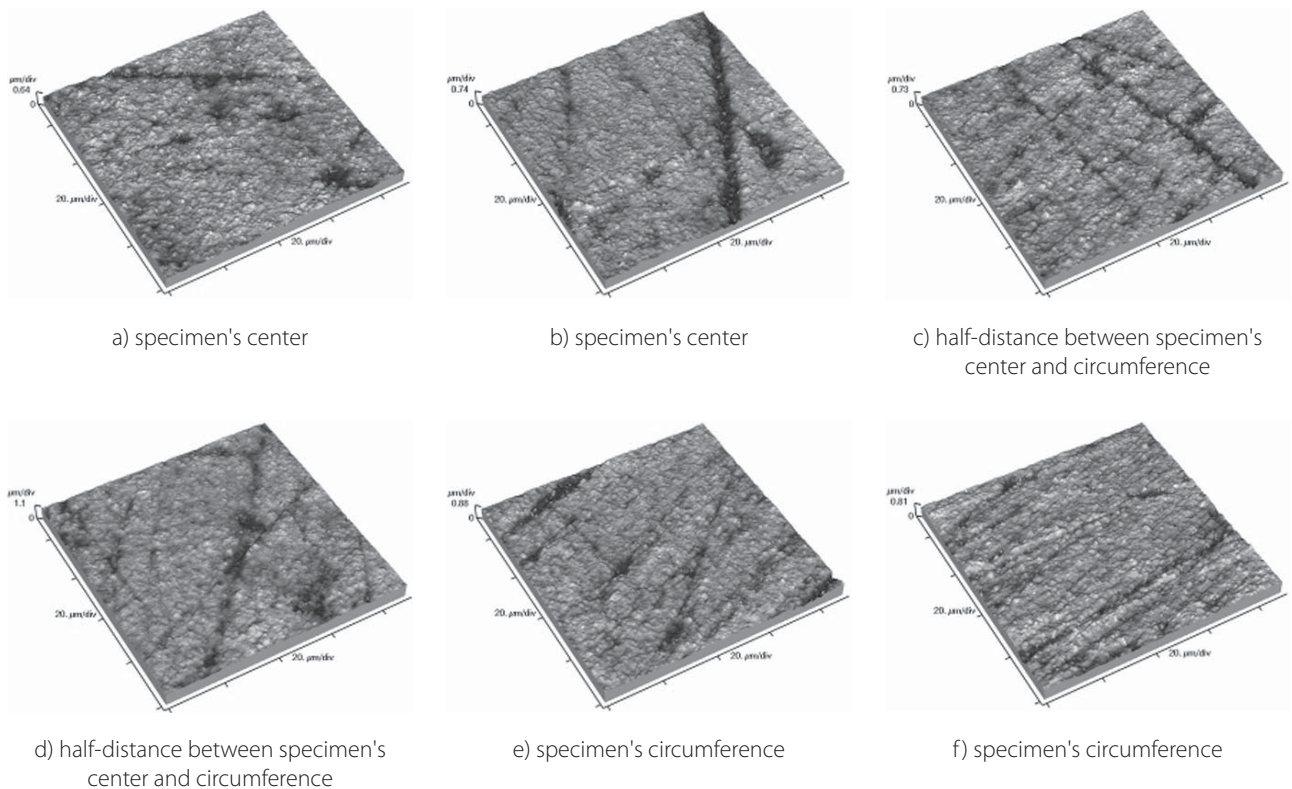


FIGURE 10. Topography of Filtek Z250 sample

(Figure 9) and similar is on Filtek Z550's surface (Figure 5). Moreover, by analyzing topography of Gradia Direct, regions with flat surfaces can be distinguished, which don't

appear on any other material in this study. Surface of Filtek Z250 is featured by individual deep channels and less visible polishing tracks and local damages (Figure 10) which are

not channel-like type. Filtek Ultimate Body has small polishing tracks without any defects on measuring points on the specimen's center (Figure 7a and b) and with deep channels and defects on measuring points close to specimen's circumference (Figure 7c, d, e, f). Filtek Ultimate Translucent demonstrated a uniform surface quality regardless of the measuring point (in the center, half-distance between center and circumference and at circumference of the specimen), which is confirmed by very close values of (R_a) and (R_{p-v}) on all of the measuring points. Measured roughness of all materials used in this study was lower than 100 nm.

DISCUSSION

After finishing and polishing procedure, all the materials had similar average surface roughness (R_a) which indicates that the used abrasive disks successfully reduced the average roughness of the materials on the relatively equal value. The lowest (R_a) values of Filtek Ultimate Body can be explained by material filler composition. This material is a nanofilled composite, filled only with nanometer size particles, from which some are dispersed and others create nanoclusters, as secondary formed fillers [12]. The size of these nanoclusters can range from about 0.6 to 10 μm , from which the polishing procedure usually plucks out the individual primary nanoparticles, and not a whole nanocluster [12, 21, 22]. Although rare, there is a possibility of the whole nanocluster dislodgement from the surface layer of the material, which can be seen as a nearly rounded surface defect that is not channel or track-shaped [19]. The highest (R_a) value of Filtek Z550 can be explained by the fact that this material is a nanohybrid composite, composed of micrometer particles, whose size range up to 3 μm , and of very small nanometer particles of 20 nm. The existence of larger micrometer particles in this material causes either protruding or dislodgement of fillers from the surface layer during polishing, remaining residual surface irregularities and defects [21, 23]. Consistency of the measured results of Tetric EvoCeram can be explained by very high percentage of inorganic filler (82.5 vol%) in this material, and it's optimal composition of heterogeneous filler particles, which provide this uniformly behavior of the material. Although Tetric EvoCeram belongs to the same material type as Filtek Z550, these materials differ in the volumetric filler proportion. In terms of uniformity of results, nanofilled composites also stood out, noting that Filtek Ultimate Translucent showed more uniform (R_a) values than Filtek Ultimate Body. The only differences, which could be indicated between these two materials, are: the size of the clusters, which are about 20 μm in Translucent, and about 10 μm in Body material; and a slightly lower volume percentage of filler in Translucent

(63.3 vol % in Body, 55.6 vol % in Translucent). It is possible that these double-sized clusters in Translucent favor more homogeneous distribution of particles in the composite, so that it implies such uniform (R_a) results of this material. Higher (R_{p-v}) values in Filtek Z550, Filtek Z250 and Filtek Ultimate Body and deep channels and polishing tracks are expected, because these are hybrid materials, whose larger microparticles protrude from the polished surface or are pulled out by abrasive disk leaving surface voids [24]. Large differences in the profile (R_{p-v}) of Filtek Ultimate Body are showing a certain heterogeneity of distribution of particles in the material and the existence of segments that are, more or less, susceptible to cleavage. It is important to emphasize that the (R_{p-v}) is a relative indicator of surface roughness and that it does not show absolute values of height or depth of channels in the material. Qualitative analysis of 3D AFM images confirmed that Tetric EvoCeram has highly-uniform surface quality on all of the measuring points (Figure 6), such as the roughness analysis found. Polishing damages on GC Gradia Direct's surface can be explained by a wear process of this microfilled material, whose surface resistance to abrasive disc treatment is lower. This is directly related to the size of filler particles and it's relatively poor capability for volumetric loading, which leaves areas with weak and soft, low-filled resin [25]. On the other hand, although nanofills are similarly volumetrically filled like microfills, they contain nanoparticles which have the unique physicochemical properties [12, 26]. Those properties are not only the matter of particle size, but also of the qualities that this small size particles cause [12, 26]. Thus, they have reinforcing influence on material that comprises them, making high surface resistance to wear caused by abrasive disc [12]. Unique flat surfaces on Gradia Direct can be explained by the existence of pre-polymerized fillers, taking as much as 42% by volume in the material. The "flat fields" on the AFM images can be explained by the fact that the pre-polymerized fillers have improved bonding properties and that almost any of the particles in this field do not pluck out during polishing procedure [19]. The disadvantage of this material is relatively low wear resistance, compared to nanofilled materials, and therefore reliability of polished surface could be short [25]. The local damages on the surface of Filtek Z250 and on Filtek Z550, which are not channel-like type, are probably caused by plucking out of the entire microparticles during polishing process and by remaining the residual surface defects, which were verified by (R_a) and (R_{p-v}) values [12]. Filtek Ultimate Body and Filtek Ultimate Translucent although two very similar nanofilled materials, represented differences in surface appearance on measuring points, depending on the location of the measurement. Through repetitive movements during polishing process, abrasive disc was falling off from the specimen's circumference which could cause an

engraving of the edge of abrasive disc into the material sample. The only difference between Filtek Ultimate Body and Filtek Ultimate Translucent is in terms of nanocluster size, which is 0.6-10 μm for Body and 0.6-20 μm for Translucent material. Remaining particles are totally the same. It could be possible that larger clusters favor Filtek Ultimate Translucent in terms of polishing properties and wear resistance caused by abrasive disc whether it is in the specimen's center or circumference. It is important to emphasize that rough surfaces favor bacterial adhesion and biofilm formation on the teeth and restorations, which can further cause secondary caries, gingival and periodontal diseases [17, 27]. There are no agreed reference data on the limit roughness below which the bacteria would not adhere [28]. The most commonly mentioned limit is below 0.2 μm for adherence of dental biofilm [17, 27]. Maybe it is most accurate to say, that it depends on the bacteria species. Roughness in this study, lower than 100 nm for all material types, is far from the mentioned limit.

CONCLUSION

Within the limitation of this study, it can be concluded that the type, size, distribution of fillers and filler loading of all of the tested materials, didn't affect average surface roughness of the samples after finishing and polishing procedure. On the other hand, different material compositions affected the topography of the polished surfaces of the materials. We should be careful when using the prefix nano-, when it comes to nanohybrids, because the overall material properties depend on all kinds of filler particles, their volume fraction and distribution. Only materials that have the optimal composition of heterogeneous filler particles, which provide their consistent behavior, or materials that consist of all particles in nanometer dimensions, allow more uniform surface topography after polishing treatment. Multi-step polishing abrasive discs, left channels on all materials regardless of composition. It is recommended for future studies to examine whether other polishing systems would leave fewer traces on polished samples.

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DECLARATION OF INTEREST

There are no scientific or financial conflicts of interest.

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