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Surface Roughness in Ceramics with Different Finishing Techniques Using Atomic Force Microscope and Profilometer

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Clinical Relevance

Ceramic restorations often require intraoral adjustment and the use of a polishing kit is mandatory to re-establish surface smoothness. When the ceramic surface was ground and polished, the 3 types of ceramic restorations reacted differently to each tested polishing kit. Some of the polished surfaces obtained were at least equivalent to glaze-fired ceramic surfaces.

SUMMARY

This study assessed the finishing and polishing of 3 ceramic materials: Vitadur Alpha, IPS Empress 2 and AllCeram. Surface modification techniques were selected to simulate dental practice. Forty-five specimens of each ceramic were divided into 5 groups of 9 specimens, which were finished using the following procedures: Group 1–glaze; Group 2–glaze, grinding and subsequent polishing with the Eve system; Group 3–glaze, grinding and subsequent polished with the Identoflex system; Group 4–glaze followed by polishing with Identoflex; Group 5–glaze, grinding and subsequent polishing with Shofu kit. Two roughness-measuring instruments were used: a stylus profilometer and an atomic force microscope (AFM). The 135 specimens were evaluated quantitatively with respect to Ra (average roughness) and Ry (maximum roughness height), and the results were examined statistically by ANOVA and Tukey's test, with a significance level of 0.05. The roughness parameter (Ra), measured by the profilometer, and AFM showed that some of the commercial intraoral polishing kits tested achieved a finish equal in smoothness to the glazed surface. According to Pearson's test, no correlation was found between the parameter Ry, measured with the profilometer, and AFM. The profilometer results for Ry demonstrated no significant differ-

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ences between the final polished surfaces and the initial glazed ones. On the other hand, the Ry values obtained by AFM indicated the tested polishing kits incapability of producing smoothness comparable to the glazed surfaces.

INTRODUCTION

Ceramic restorations can provide the most natural replacements for teeth, and the use of ceramic materials in dentistry has recently increased, extensively due to a high rate of esthetic demand by patients (Kawai, Urano & Ebisu, 2000). As with any dental restorative material, certain potentially adverse biological effects must be appreciated (Ward, Tate & Powers, 1995). The oral cavity is constantly contaminated by many diverse microbial species, most of them, especially those that are responsible for caries and periodontitis, can only survive in the mouth when they adhere to non-shedding surfaces. Surface-free energy can play a role in bacterial adhesion and retention; however, according to Quirynen and Bollen (1995), the influence of surface roughness overrules surface-free energy. Rough surfaces may also lead to abrasion of adjacent and opposing teeth and tooth staining (Jagger & Harrison, 1994). Therefore, the surface roughness of restorative materials should be minimized to contribute to the patient's comfort, optimum esthetics, oral hygiene and guarantee clinical success.

Although previous studies have stated that ceramics exhibit the least amount of bacterial and glucan adhesion compared to amalgam, resin composite and casting alloy (Kawai & Urano, 2001), explaining the fact that gingival tissues respond best to this material when preparing a ceramic crown for final placement into a patient's mouth, the grinding process breaks the thin glaze surface layer and requires it to be reglazed or, alternatively, polished.

Many authors studied the effect of polishing techniques on the surface roughness of ceramic restorations, but the results were controversial (Sulik & Plekavich, 1981; Ward & others, 1995; Chu, Frankel & Smales, 2000; Pattersson & others, 1992; Bessing & Wiktorsson, 1983; Scurria & Powers, 1994). It is clear that roughness is an important fact to be considered and, in scientific literature, several methods have been employed in order to characterize a surface with respect to its roughness. These methods include contact stylus tracing, laser reflectivity, non-contact laser stylus metrology, scanning electron microscopy and compressed air measuring (Leitão & Hegdahl, 1981; Haywood, Heymann & Scurria, 1989; Whitehead & others, 1995). The fact that roughness does not appear with a certain pattern and multiple roughness scales contribute to both the complexity of measuring and studying surface roughness, the presence of voids and irregularities makes accurate measuring of surface roughness in ceramics even more difficult (Ward & others, 1995). That is why the literature suggests that surface characteristics should be described as using more than 1 surface measurement parameter, such as Ra, the arithmetical average value of all absolute distances of the roughness profile; Ry, the maximum peak to valley distance; Rz, the average maximum peak to valley height or Rpm, the mean value of leveling depths of 5 consecutive sampling lengths, in order to give reliable information on the profile shape (Whitehead & others, 1995).

With the increased use of ceramic materials in dentistry, one must pay careful attention to the efficacy of different polishing systems and techniques, the brittle nature of this material and whether or not the intraoral polishing techniques, using rotary instruments such as diamond burs and rubber abrasives, can provide a clinically acceptable smoothness due to the high hardness of ceramic materials. This study evaluated the surface roughness of 3 different brands of ceramic materials when modified by different intraoral polishing procedures, as compared to a thermally-glazed control group. The specimens were quantitatively evaluated with a profilometer and an atomic force microscope with respect to Ra (average roughness) and Ry (maximum peak to valley height).

METHODS AND MATERIALS

One hundred and thirty-five ceramic disks 5-mm in diameter and 3-mm thick, were fabricated according to the manufacturer's recommendations. Table 1 lists the ceramic materials used in this study. The abrasive instruments used to finish and polish the ceramic samples are described in Table 2.

Table 1: *Ceramic Materials*

Ceramic	Company
AllCeram	Degussa
IPS Empress 2	Ivoclar Vivadent, Schaan, Liechtenstein
Vitadur Alpha	Vita Zahnfabrik GmbH, Germany

Table 2: *Instruments and Polishing Kits*

Instruments and Polishing Kits	Company
Diamond burs 3195, 3195 F and 3195 FF	KG Sorensen
Shofu (Ceramiste points: Standard, Ultra and Ultra II)	Shofu Inc, Kyoto, Japan
Identoflex (N 7060 and N 7260 Rubber points)	Identoflex, Buchs SG, Switzerland
Eve (RA 105 Diamond, polisher points W16Dg, W16Dmf, W16D)	Eve, Ernst Vetter, GmbH, Germany

The samples of each different material were divided into 5 groups of 9 samples. Each group received a different surface treatment. To simulate clinical procedure, the grinding and polishing steps were carried out manually by a single operator (Jung, 2002; Fuzzi, Zaccheroni & Vallania, 1996; Pattersson & others, 1992; Ward & others, 1995; Chu & others, 2000). The ground samples were abraded under water cooling with progressively smaller particle sizes: 90 µm diamond, followed by a 38 µm and a 25 µm for 5 seconds each (Haywood & others, 1989). The polishing technique, including cooling and revolutions per minute (RPM), were set according to the manufacturer's recommendations and used for 30 seconds each; the polishing material was used by a single operator, who applied uniform pressure and uniform application time for each surface treatment (Ward & others, 1995). The surface treatment of each group is summarized in Table 3.

Table 3: *Specimen Groups Classified in Accordance to the Surface Treatments*

Group	Oven Glazed	Surface Treatment
1	Yes	Control
2	Yes	Ground + Polished (Eve)
3	Yes	Ground + Polished (Identoflex)
4	Yes	Polished (Identoflex)
5	Yes	Ground + Polished (Shofu)

In Group 1, the surface was left as glazed (control); in Group 4, the original glaze was over-polished using a finishing kit to simulate the effect of extending the refinishing onto the original glaze at the periphery of a surface of adjusted ceramic (Pattersson, 1991).

Roughness measurements were performed on each disk using a profilometer (SURFTEST-SV-600 s 178 Surface Roughness Tester, MITUTOYO, Co, Santo Amaro, São Paulo, Brazil) with a cut-off value of 0.25 mm. Three different regions were evaluated in each specimen to determine 3 Ra and Ry values and the final value to characterize that each sample was the arithmetic mean among them.

A MultiMode atomic force microscope (Afm Nanoscope IIIa-Veeco Instruments, Santa Barbara, CA, USA), operated in contact mode, was used to obtain a quantitative and qualitative evaluation of the samples. In contact mode, the force between the AFM tip and the sample surface was kept constant by the microscope feedback system, while the sample surface was scanned beneath the AFM tip and the vertical piezoelectric ceramic movement was recorded. Images with 512 x 512 pixels were acquired with a scan size of 20 µm x 20 µm and a scan rate of 2.03 Hz. An NP-type V-shape Si_3N_4 cantilever (Veeco Instruments, Santa Barbara, CA, USA) with a normal bending constant of k=0.075 N/m and a tip radius of approximately 100 nm was used. During imaging, the set point was chosen to be 2.0 volts higher than the top-bottom laser photo detector output obtained when the tip was out of surface contact. The AFM obtained a 3-dimensional image of the surface of the samples. Again, 3 different areas were measured in each sample at different regions, all located in the center of the samples. As it was performed with the other instrument, the measured Ra and Ry values determined one final value for each parameter, which was the arithmetic mean among them. It has to be noted that, before any surface roughness evaluation, the AFM images were first processed with a linear plane fit to remove any sample tilt in respect to the AFM tip and, secondly, by flattening the images in order to adjust the average heights of the scanned lines in the AFM image. The surface roughness parameters, Ra and Ry, were then calculated in both slow and fast scanning directions with the use of Nanoscope software built-in functions. Means and standard deviations of surface roughness from this equipment were determined. Data were analyzed by a 2-way analysis of variance (ANOVA) for each ceramic material and polishing method. Tukey intervals were calculated from analysis of variance at the 0.05 significance level for comparisons of means among the ceramics and surface treatments. A Pearson's test was carried out to investigate the correlation between the profilometer and AFM surface roughness data.

RESULTS

Test Carried Out by a Profilometer

Mean Ra values and standard deviation of the 3 ceramic materials after different surface treatments are shown in Figure 1. The obtained values of Ra showed statistically significant differences in surface roughness among surface treatments. IPS Empress 2 was smoother in Groups 1, 3, 4 and 5 than when ground and polished by the Eve system (Group 2). Ceramic Vitadur Alpha was significantly smoother in Groups 1, 2, 4 and 5 than in Group 3 (polished by Identoflex system). AllCeram was smoother in Groups 1, 2, 4 and 5 than when polished by the Identoflex system (Group 3). The obtained mean values of Ra showed significant differences among the ceramic materials too, as the Eve system produced smoother surfaces in AllCeram and Vitadur Alpha ceramic compared to IPS Empress 2.

Mean Ry values and standard deviations are shown in Figure 2. Multiple analyses of variance showed significant differences that were statistically significant between surface treatments and the materials tested; the ceramic AllCeram, ground and finished by the Identoflex system (Group 3), had a significantly rougher surface than in Groups 1, 2, 4 and 5 and rougher values than IPS Empress 2 and Vitadur Alpha specimens when they received the same surface treatment (Group 3). For IPS Empress 2 and Vitadur Alpha ceramics, there was no significant difference among the tested treatments.

Test Carried Out by an Atomic Force Microscope

Mean Ra values and the standard deviation of ceramics and surface treatments are shown in Figure 3. The Eve polishing system (Group 2) was the least effective system tested on IPS Empress2 ceramic, followed by the Identoflex system (Group 3); the glazed surface had no significant difference compared to Groups 4 and 5. For the Vitadur Alpha ceramic, the glazed surface (Group 1) was smoother when compared to Groups 5 and 2, with no statistical difference to Groups 3 and 4. The AllCeram material was rougher when polished by the Identoflex system (Group 3) compared to Groups 1, 2 and 4; on the other hand, when polished by Shofu (Group 5), it was rougher than Groups 1 and 4. For the materials tested, IPS Empress 2 presented higher roughness values than Vitadur Alpha and AllCeram in Group 2. In Group 3, IPS Empress 2 also presented higher roughness than Vitadur Alpha ceramic.

Mean Ry values and standard deviation of the surface roughness of ceramics and surface treatments are shown in Figure 4. The glazed surfaces produced smoother surfaces for all 3 brands of ceramics. The glazed surface that was subsequently polished without grinding (Group 4) was the next smoother surface observed in all 3 ceramic materials. IPS Empress 2 surface polished with the Eve system (Group 2) was rougher than AllCeram and Vitadur Alpha polished the same way.

When the Identoflex system was used (Group 3), Vitadur Alpha had a smoother surface than AllCeram. Vitadur Alpha ceramic presented no significant difference among Groups 1, 3 and 4. It is relevant to point out that all the comparisons given are statistically valid at a significance level 0.05 and that the images obtained from the Atomic Force Microscope corroborate the measured values previously verified in the AFM (Figures 5, 6 and 7).

DISCUSSION

Solid surfaces can be formed by either of the following methods: fracture of solids or machining, such as grinding, thin-film deposition and the solidification of liquids. It is found that most solid surfaces formed by these methods are not smooth. Perfectly flat surfaces that are

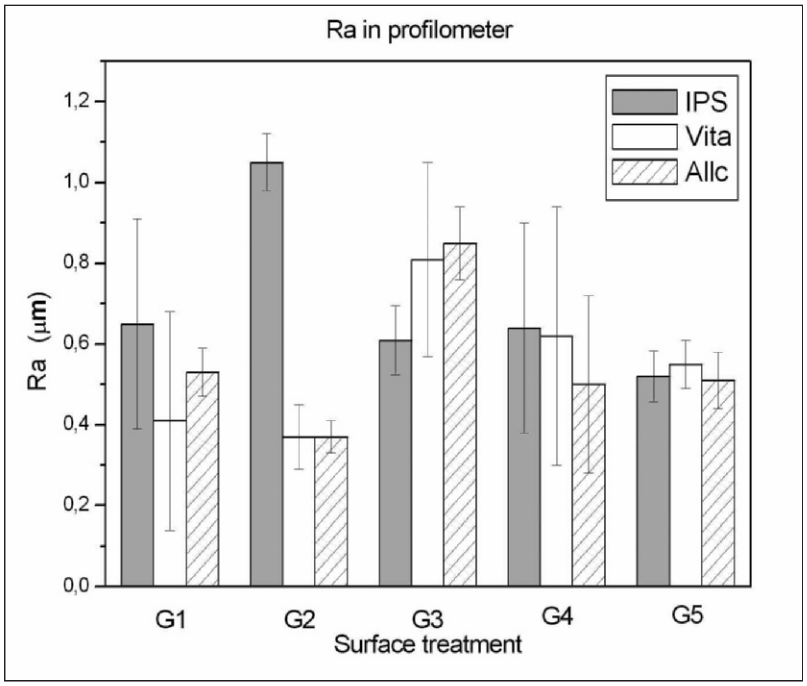

Figure 1: *Mean and standard deviation of Ra values in Profilometer. 1–glaze; 2–glaze, grinding and subsequent polishing with Eve system; 3–glaze, grinding and subsequently polished with Identoflex system; 4–glaze followed by polishing with Identoflex; 5–glaze, grinding and subsequent polishing with Shofu kit.*

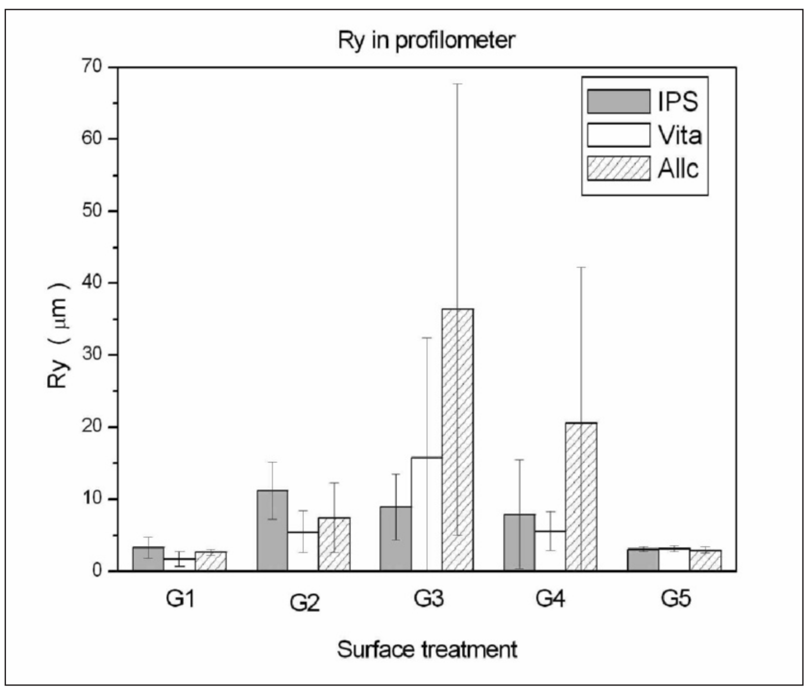

Figure 2: *Mean and standard deviation of Ry values in Profilometer. 1–glaze; 2–glaze, grinding and subsequent polishing with Eve system; 3–glaze, grinding and subsequently, polished with Identoflex system; 4–glaze followed by polishing with Identoflex; 5–glaze, grinding and subsequent polishing with Shofu kit.*

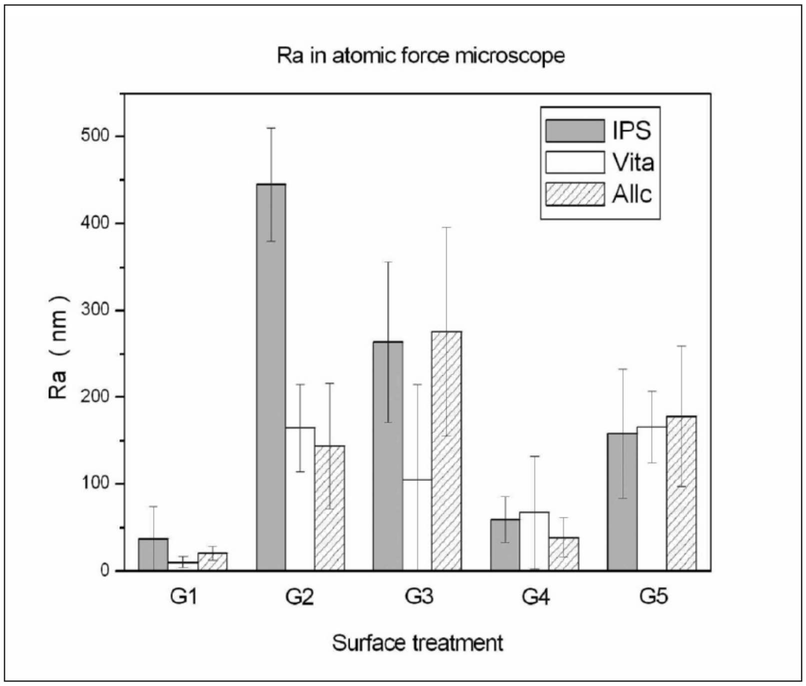

Figure 3: *Mean and standard deviation of Ra values in AFM. 1–glaze; 2–glaze, grinding and subsequent polishing with Eve system; 3–glaze, grinding and subsequently polished with Identoflex system; 4–glaze followed by polishing with Identoflex; 5–glaze, grinding and subsequent polishing with Shofu kit.*

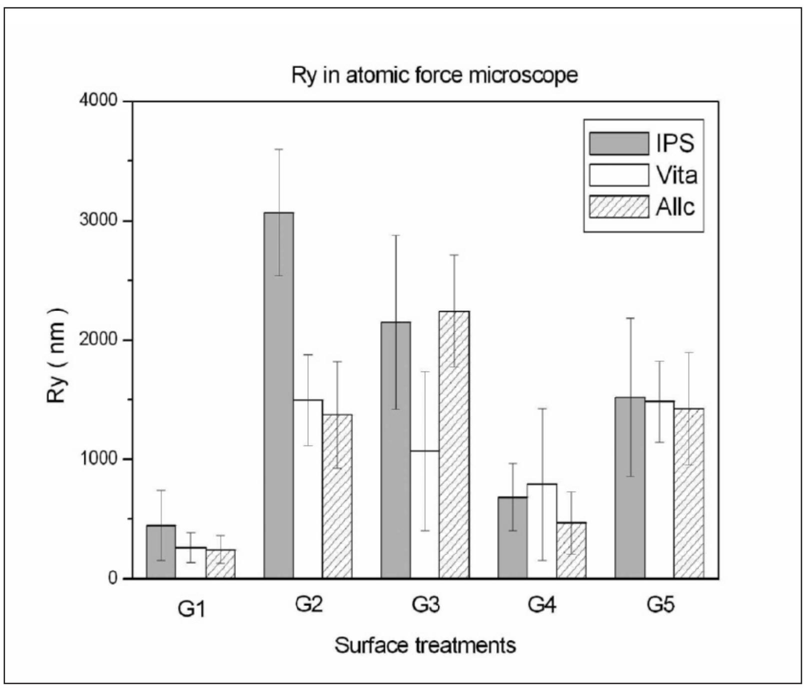

Figure 4: *Mean and standard deviation of Ry values in AFM. 1–glaze; 2–glaze, grinding and subsequent polishing with Eve system; 3–glaze, grinding and subsequently polished with Identoflex system; 4–glaze followed by polishing with Identoflex; 5–glaze, grinding and subsequent polishing with Shofu kit.*

smooth, even on the atomic scale, can be obtained only under very carefully controlled conditions and are very rare (Majumdar & Bhushan, 1995). Surfaces can be smooth, such as the Himalayas viewed from space, but the same surface can also be rough, much like the same mountains viewed from earth. In general, morphology depends on the length scale of observation. Surface irregularities viewed with magnification may go unnoticed when examined with the unaided eye (Barabási & Stanley, 1995). How can we describe a surface that is smooth to the eye but rough under a microscope? The fact is that roughness changes when the observation scale itself changes. This was detected in this investigation and related by other authors who have compared the results of surface roughness obtained by different measuring methods (Sulik & Plekavich, 1981; Klausner, Cartwright & Charbeneau, 1982; Bessing & Wiktorsson, 1983; Ward & others, 1995; Fuzzi & others, 1996). One of the prime requisites for a satisfactory ceramic restoration is a highly smooth surface, which contributes to the patient's comfort, optimum esthetics, oral hygiene and low plaque retention (El-Karaksi, Shehab & Eskander, 1993).

This study evaluated the efficacy of different ceramic intraoral finishing kits; for this purpose, 2 different types of equipment have been used: an atomic force microscope and a classic method, a profilometer (Whitehead & others, 1995).

The controversy concerning the effectiveness of polishing so as to obtain an acceptable smooth ceramic surface is still current. Authors have published the fact that polishing systems can obtain a final ceramic surface that is at least equivalent to glaze-fired ceramic surfaces, if not better (Sulik & Plekavich, 1981; Klausner & others, 1982; Bessing & Wiktorsson, 1983; Haywood & others, 1989; Scurria & Powers, 1994; Ward & others, 1995). Other researchers have denied this (El Karaksi & others, 1993; Chu & others, 2000; Pattersson & others, 1992). The results have shown that adjusting ceramic surfaces, which cannot later be reglazed, should be avoided, as nearly none of the polishing procedures tested will recreate a surface similarly smooth to that of the original glaze (Pattersson & others, 1992). The authors of this study agree with Klausner and others (1982) that the use of an AFM, SEM or intraoral macroscopic lens is obviously not feasible for the clinician. These lenses are, however, helpful in the laboratory evaluation and development

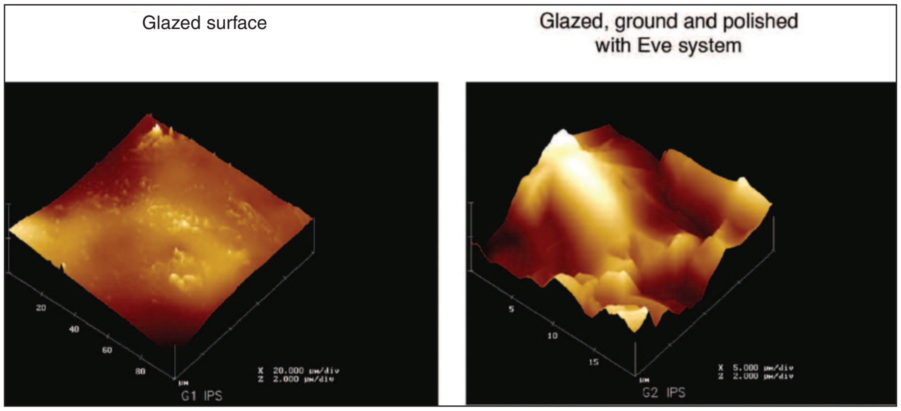

Figure 5: *Atomic force microscope images—IPS Empress 2 Ceramic.*

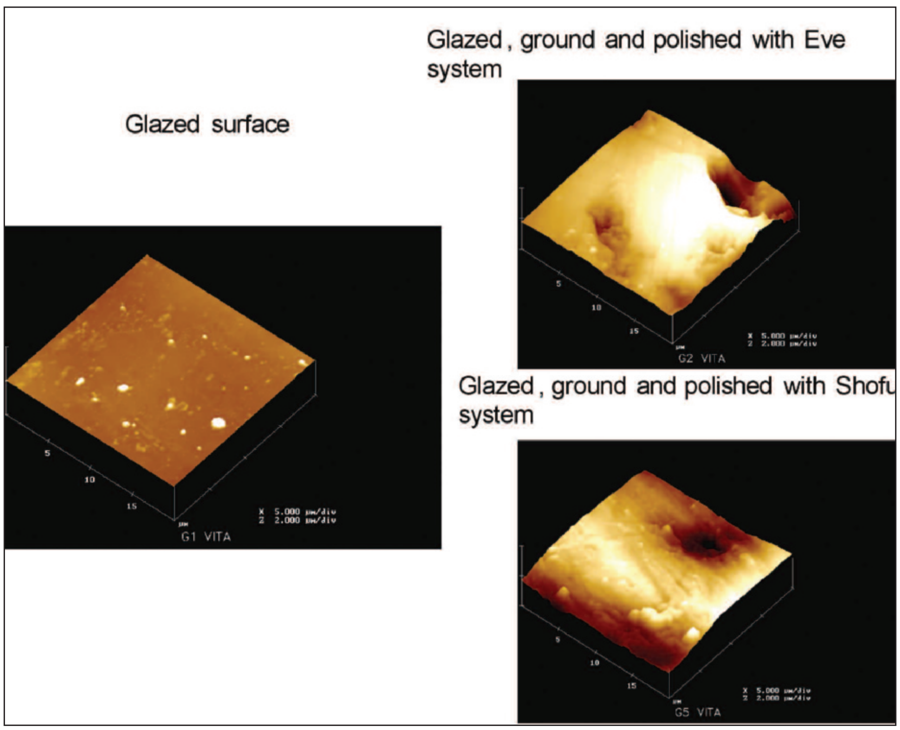

Figure 6: *Atomic force microscope images—Vitadur Alpha Ceramic.*

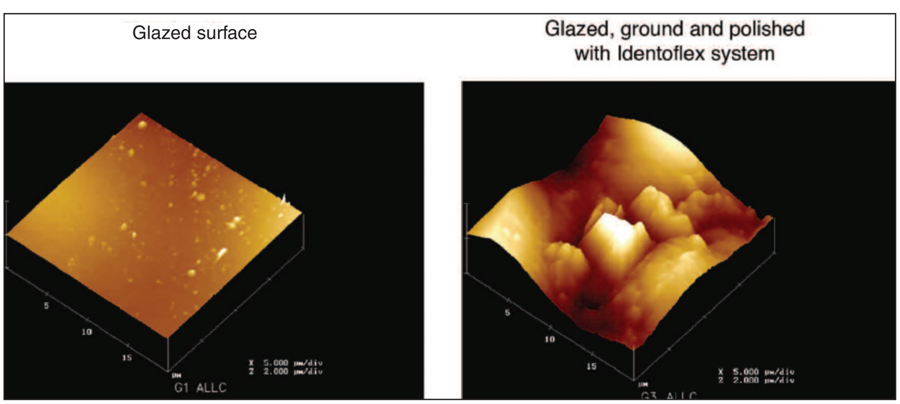

Figure 7: *Atomic force microscope images—All Ceram.*

of polishing abrasives and sequences. The use of an atomic force microscope is an alternative method, with high resolution in nanometer scales (Marshall & others, 2001a,b; Mannelquist, Almquist & Fredriksson, 1998; Arvidsson, Milleding & Wennerberg, 2002). While the AFM covers a square area, the profilometer determines the profile along 3 lines on the surface by the use of a tracing device, and it expresses roughness by the undulations of the profile relative to some base line. Although the AFM verifies a smaller area, it is representative, because it goes point by point and covers the whole. As the roughness is scale-dependent and increases when a larger area is studied, the higher values observed in the profilometer were already expected (Leitão, 1982). The AFM holds significant promise for the study of biomaterials, as it has some important advantages such as minimal sample preparation, high resolution and visualization of a 3-dimensional image of the surface (Hegedüs & others, 1999; Yu & Namba, 1998). Furthermore, it allows for reexamination of the same specimen. The current application of this instrument for measuring the surface roughness of ceramics or other dental materials still depends on establishing protocols, which is why the profilometer is still the classic method.

As far as the Ra parameter is concerned, the profilometer and the atomic force microscope have shown similar patterns; they have both demonstrated that some polished surfaces had no significant difference compared to the initial glazed surface. On the other hand, when one considers the Ry values, the profilometer demonstrated that, apart from the Identoflex system used in the AllCeram material, all the other kits tested obtained a smooth surface, comparable to the glazed one. In the atomic force microscope, the Ry values revealed that all the polishing systems tested were incapable of restoring a smoothness comparable to the glazed surface. The over-polished experimental group showed no significant difference compared to the glazed surface in both instruments. The authors of this both instruments. The authors of this study agree with some authors who have theorized that some profilometer readings of the ceramic surface may be misleading (Bessing & Wiktorsson, 1983) and that the presence of voids on a ceramic surface makes measuring the surface roughness of this material very difficult. In this investigation, defects were present in the AFM images of the surfaces representing voids from incomplete condensation of the ceramic material, which were also detected in other studies (Sulik & Plekavich, 1981; Fuzzi & others, 1996; Haywood & others, 1989). The controversial results of the Ry values of this study can be explained, as they are scale-dependent and influenced by the measurement technique. Instruments with different scale sensitivity may show different results.

Although different bandwidths were detected by the 2 different local probe instruments, well-correlated results of Ra have been obtained, agreeing with Vatel and others (1993). Technical literature contains papers that discuss how the results obtained for roughness measurements are affected by several factors. Some pertain to the material itself, the presence of voids, the degree of filtration and cut-off (used as a means of separating or filtering the wavelengths of a component and its numerical value determined by the ISO norm) used (Arvidsson & others, 2002). It becomes obvious that a numerical characterization of surface roughness, to a large extent, depends on the roughness parameter chosen and the measuring equipment (Leitão, 1982). By combining AFM and optical profilometer measurements, one can characterize the surface topography over a range of length scales from 0.2 µm to 4 mm (Chauvy, Madore & Landolt, 1998). The results have shown that ceramic materials can be polished to different degrees of smoothness when submitted to different systems, agreeing with Bollen, Lambrechts and Quirynen (1997), who have stated that the range in surface roughness of different intraoral hard surfaces is wide, and the impact of dental treatments on surface roughness is material-dependent, indicating that every dental material needs its own treatment modality in order to obtain and maintain the surface as smoothly as possible.

It must be pointed out that the definition of topography needs to be considered in relation to issues concerning the smoothness of restorations in order to be clinically acceptable (Hulterström & Bergman, 1993; Willems & others, 1991; Jung, 2002; Türkün & Türkün, 2004). The main controversy is that, on one hand, some *in vivo* studies (Bollen & others, 1997) suggest an ideal threshold surface roughness for bacterial retention (Ra = 0.2 µm). On the other hand, enamel roughness is also reported to be a guideline parameter, but it depends on the tooth type and location in the oral cavity. If tooth enamel roughness is regarded as a guide parameter of Ra, the authors can interpret the results of this *in vitro* study as clinically acceptable, despite statistical differences.

CONCLUSIONS

1. The 3 brands of ceramic materials tested reacted differently when submitted to the same polishing procedure.
2. The scale-dependent Ra and Ry values were influenced by the measurement technique.
3. New protocols must be determined to study surface roughness in ceramics with an atomic force microscope.
4. By combining the Profilometer and Atomic Force Microscope, the surface topography can be characterized over a length scale from 0.01 µm to 4 mm and, consequently, the results are more reliable and precise.
5. The authors recommend avoiding adjusting ceramic restorations after they have been placed in the mouth and, above all, when using Vitadur Alpha ceramic. When IPS Empress 2 ceramic restorations are used and the execution of grinding procedures are imperative, clinicians should choose either a Shofu or an Identoflex system in order to achieve acceptable results. As to AllCeram ceramic restorations, in case grinding is inevitable, either the Shofu or Eve system should be selected.

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