Evaluation of Spatial and Functional Roughness Parameters on Air-abraded Zirconia as a Function of Particle Type and Deposition Pressure

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Purpose: This study evaluated the spatial and functional roughness parameters on air-abraded zirconia as a function of particle type and deposition pressure.

Materials and Methods: Polished zirconia blocks (Cercon, Degussa/Dentsply) (N=30) with dimensions of 5 × 4 × 4 mm^3 were air abraded according to 2 factors: a) particle type – 30-µm silica-coated alumina (CoJet) or alumina particles (45 µm); b) deposition pressure (1.5, 2.5 and 4.5 bar). Roughness parameters (S_{dr}, V_i, S_{ci} and S_{vi}) were measured in an optical profilometer (Wyko NT 1100) at the center of the air-abraded area (301.3 × 229.2 µm). Two measurements were made for each parameter from each surface. The means of each group were analyzed by 2-way ANOVA followed by Tukey's adjustment test and Student's t-test (alpha = 0.05).

Results: Both the particle type ($p < 0.05$) and deposition pressure ($p < 0.05$) significantly affected the roughness parameters. Interaction terms were significant except for S_{ci} and S_{vi}. With the increase in pressure from 1.5 to 4.5 bar, S_{dr} (CoJet 1.5: 15.7 ± 0.2; CoJet 4.5: 26.6 ± 0.2; alumina 1.5: 14.7 ± 0.2; alumina 4.5: 24.4 ± 0.2) and V_i (CoJet 1.5: 0.66 ± 0.01; CoJet 4.5: 1.37 ± 0.07; alumina 1.5: 0.62 ± 0.02; alumina 4.5: 1.19 ± 0.02) parameters showed a significant increase with both alumina and CoJet particles. Mean S_{ci} values (CoJet 1.5: 1.62 ± 0.01, CoJet 4.5: 1.49 ± 0.02; alumina 1.5: 1.6 ± 0.03; alumina 4.5: 1.42 ± 0.04) and S_{Vi} (CoJet 1.5: 0.98 ± 0.01, CoJet 4.5: 0.112 ± 0.01; alumina 1.5: 0.98 ± 0.01, alumina 4.5: 0.12 ± 0.01) decreased significantly ($p < 0.05$) with the increase in pressure from 1.5 to 4.5 bar. The pressure increase from 2.5 to 4.5 bar did not cause any significant difference ($p > 0.05$) in these parameters for either particle type.

Conclusion: Considering roughness parameters for micromechanical retention and parameters for adsorption mechanisms of adhesion, zirconia surfaces presented better morphological features when air abraded with silica-coated alumina than alumina particles at pressures higher than 1.5 bar. Particle deposition at 2.5 bar may be preferable to 4.5 bar pressure for avoiding possible deposition-related damage on zirconia, as there were no significant differences for the functional parameters.

Keywords: adhesion, ceramic, prosthodontics, surface treatment, roughness, zirconia.

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Surface conditioning methods based on particle deposition, namely air-borne particle abrasion using only alumina or silica-coated alumina on zirconia, are intended to remove surface contaminants, increase bonding surface area to promote micromechanical interlocking, improve adhesion of resin to this ceramic, expand wetting kinetics, and increase surface energy.[2,4,6] A variety of surface roughness parameters exist, but average roughness (R_a) is the most commonly studied roughness parameter for comparative analysis between adhesion promoters and micromechanical retention for zirconia in the dental literature.[2] In fact, the R_a parameter provides limited information on surface outline and could result in misleading conclusions when used alone to establish the actual

Table 1 Results of 2-way ANOVA for S_{dr}, V_i, S_{ci} and S_{vi} parameters considering the factors particle size (A) and deposition pressure (B) ($p < 0.05$)

Factor	S_{dr}	V_i	S_{ci}	S_{vi}
A	0.0	0.0	0.0001	0.0268
B	0.0	0.0	0.0	0.0
A × B	0.0008	0.0001	0.0714	0.2681

correlation between zirconia surface roughness and adhesive bond strength.[3] Thus, complimentary to the 2D conventional roughness parameters, spatial and functional roughness parameters could provide additional information for interpreting physicochemical interactions between zirconia and adhesive materials.[2,3]

In this context, S_{dr} (surface area ratio) indicates the increment of the interfacial surface area related to the area of the projected xy plane,[3] and V_i (volume of interdigitation) estimates the apparent volume of interdigitation available on the surface.[1] In addition, S_{ci} (core fluid retention index) and S_{vi} (valley fluid retention index) are derived from the analysis of the bearing surface area, quantifying the volume of fluid filling the core and the valleys of the given surface, respectively.[3] All these parameters could be influenced by deposition-related parameters.

The objective of this study was thus to evaluate the spatial and functional roughness parameters on air-abraded zirconia as a function of particle type and deposition pressure. The null hypothesis tested was that neither the particle type nor the deposition pressure would affect the roughness parameters for zirconia.

MATERIALS AND METHODS

Specimen Preparation

Zirconia (Cercon, Degussa/Dentsply; York, PA, USA) blocks (N = 30) with dimensions of 5 mm × 4 mm × 4 mm were sintered and polished with 1200-grit abrasive papers under water cooling. They were then ultrasonically cleaned in distilled water for 10 min and randomly divided into 6 groups (n = 5/group) according to 2 factors: a) particle type – 30-µm silica-coated alumina (CoJet, 3M ESPE; Seefeld, Germany) or alumina (45 µm, Polidental; São Paulo, SP, Brazil) and b) deposition pressure (1.5, 2.5, and 4.5 bar). Air abrasion was performed in the same location for 2 s at a distance of 10 mm perpendicular to the surface.[7]

Roughness Measurements

Roughness parameters (S_{dr}, V_i, S_{ci}, S_{vi}) were measured in an optical profilometer (Wyko NT 1100, Veeco; Plainview, NY, USA). From each surface, measurements (301.3 × 229.2 µm) were made at the approximate center of the air-abraded area.

Statistical Analyses

The means of each group were analyzed using 2-way ANOVA (Statistix 8.0 for Windows, Analytical Software; Tallahassee, FL, USA) for each roughness parameter (dependent variable) and particle type (2 levels) and deposition pressure (3 levels) as the independent variables. Multiple comparisons were made by Tukey's adjustment test. p-values < 0.05 were considered to be statistically significant in all tests.

RESULTS

Both the particle type ($p < 0.05$) and deposition pressure ($p < 0.05$) significantly affected the roughness parameters. Interaction terms were not significant except for S_{ci} and S_{vi} (Table 1).

With the increase in pressure from 1.5 to 4.5 bar, S_{dr} (%) (CoJet 1.5: 15.7 ± 0.2, CoJet 4.5: 26.6 ± 0.2; alumina 1.5: 14.7 ± 0.2, alumina 4.5: 24.4 ± 0.2) and V_i (µm^3/µm^2) (CoJet 1.5: 0.66 ± 0.01, CoJet 4.5: 1.37 ± 0.07; alumina 1.5: 0.62 ± 0.02, alumina 4.5: 1.19 ± 0.02) parameters showed a significant increase with both alumina and CoJet particles (Table 2). Mean S_{ci} (CoJet 1.5: 1.62 ± 0.01, CoJet 4.5: 1.49 ± 0.02; alumina 1.5: 1.6 ± 0.03, Alumina 4.5: 1.42 ± 0.04) and S_{vi} (CoJet 1.5: 0.98 ± 0.01, CoJet 4.5: 0.112 ± 0.01; alumina 1.5: 0.98 ± 0.01, alumina 4.5: 0.12 ± 0.01) values decreased significantly ($p < 0.05$) with the increase in pressure from 1.5 to 4.5 bar, but the increase from 2.5 to 4.5 bar did not cause any significant difference ($p > 0.05$) in these parameters with either particle type (Table 3).

DISCUSSION

For the analyzed air-abrasion protocols on zirconia, both the particle type and deposition pressure significantly affected the roughness parameters. Since the interaction terms were only significant for S_{dr} and V_i parameters, the hypothesis could only be partially accepted. The literature provides little information regarding surface roughness analysis on zirconia after different air-abrasion protocols.[7] It is acknowledged that surface roughness can interfere with crack propagation during debonding or affect the voids trapped at the adhesive interfaces. Moreover, the main roughness parameter (R_a) has been demonstrated to have poor correlation with bond strength results,[4] probably because R_a provides limited information about detailed surface texture.[3]

The mechanisms used for assessing the quality of adhesion at adhesive interfaces are essentially diffusion, electrostatics, adsorption, and mechanical interlocking. Accordingly, S_{dr} can help understand the chemical/physical effect of surface conditioning on the ultimate bond strength results. This is because the adsorption mechanism is correlated to the interaction between the surface area of substrate, adhesive, and adherents, where the bond strength results also take

Table 2 Mean values (± SD) of different parameters measured on zirconia surfaces after air abrasion with two particle types under three levels of deposition pressure

Particle type	Deposition pressure (bar)	S_{dr} (%)	V_i (µm³/µm²)	S_{ci}	S_{vi}
CoJet	4.5	26.6 (0.2)[a]	1.37 (0.07)[a]	1.49 (0.02)[bc]	0.112 (0.01)[a]
CoJet	2.5	23.3 (0.5)[b]	0.96 (0.04)[c]	1.51 (0.04)[b]	0.11 (0.01)[ab]
CoJet	1.5	15.7 (0.2)[d]	0.66 (0.01)[e]	1.62 (0.01)[a]	0.98 (0.01)[b]
Alumina	4.5	24.4 (0.2)[b]	1.19 (0.02)[b]	1.42 (0.04)[d]	0.12 (0.01)[a]
Alumina	2.5	18.9 (0.3)[c]	0.77 (0.01)[d]	1.44 (0.03)[cd]	0.118 (0.01)[a]
Alumina	1.5	14.7 (0.2)[d]	0.62 (0.02)[e]	1.6 (0.03)[a]	0.98 (0.01)[b]

Different superscript letters within the same column for each roughness parameter indicate statistically significant difference (Tukey's test, *$p<0.05$).

Table 3 Mean values (± SD) of different parameters according to particle size and deposition pressure

	Particle type		Deposition pressure (bar)		
	CoJet	Alumina	1.5	2.5	4.5
S_{dr} (%)	21.9 (4.7)[a]	19.3 (4.3)[b]	15.2 (1.43)[c]	21.1 (2.35)[b]	25.5 (1.18)[a]
V_i (µm³/µm²)	1 (0.3)[a]	0.86(0.3)[b]	0.64 (0.03)[c]	0.87 (0.11)[b]	1.28 (0.11)[a]
S_{ci}	1.54 (0.06)[a]	1.49 (0.09)[b]	1.61 (0.02)[a]	1.47 (0.05)[b]	1.45 (0.05)[b]
S_{vi}	0.107 (0.001)[b]	0.112 (0.001)[a]	0.098 (0.001)[b]	0.114 (0.001)[a]	0.116 (0.001)[a]

Different superscript letters within the same row for each roughness parameter indicate statistically significant difference (Tukey's test, $p < 0.05$).

the edge of the specimens tested into consideration.[7] In this study, S_{dr} results indicated that the higher deposition pressure of 4.5 bar and 30-µm silica-coated alumina particles were more effective on zirconia surfaces than were lower pressures (2.5 and 1.5 bar) and 45-µm alumina particles.

S_{ci} and S_{vi} parameters could be relevant for the mechanical interlocking mechanism. Overall, CoJet particles presented significantly higher S_{ci} values, while alumina particles delivered higher S_{vi} values. The mechanism by which the fluid volume retention on different zones (core and valleys) of the surface affects the flow of adhesive resins and their hydrolytic stability at the interface warrants further research where these roughness parameters are correlated to bond strength results. Nevertheless, besides the physicochemical advantage provided by air abrasion with silica-coated alumina followed by silanization,[6] the surface texture using this particle type presented a higher volume of interdigitation with greater availability on the core zone (S_{ci}) than did surfaces air abraded with alumina particles. This implies that the surface features result from deposition with this particle improves micromechanical interlocking compared to alumina.[6] This, however, was the opposite for S_{vi}. The question remains to be answered whether the benefit of this kind of air abrasion is primarily micromechanical retention or physicochemical interaction with the silane treatment.

Currently, clinicians face the dilemma of whether or not to air abrade zirconia due to a possible aging effect of particles. On the other hand, sufficient adhesion must be achieved between resin cements and zirconia, especially for resin-bonded fixed dental prostheses. Since the increase from 2.5 to 4.5 bar did not cause any significant difference for the S_{ci} and S_{vi} parameters for either particle type, air-abrasion protocols at pressures above 2.5 bar may not be necessary for adequate adhesion. At this pressure, it should be considered that sandblasting using CoJet particles (30 µm) produces less monoclinic phase and residual stress in the zirconia surface than do alumina particles (50 µm).[5]

Finally, it is important emphasize that the use of 2D surface roughness parameters to predict bond strength results may not be sufficient, since maximum contact between the adherent and adhesive interface dismisses voids and flaws. Thus, the tested spatial and functional parameters in this study could provide additional information in future studies on adhesion to zirconia.

CONCLUSIONS

1. Air abrasion with silica-coated alumina particles at 2.5 bar pressure increased the surface area ratio, volume of interdigitation, and core fluid retention index on zirconia, but the valley fluid retention index was more favorable for alumina.
2. Based on the results of S_{ci} and S_{vi} parameters, particle deposition at 2.5 bar could be preferable to 4.5 bar pressure for avoiding possible deposition-related damage on zirconia, as there were no significant difference for these spatial parameters.

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Clinical relevance: Air abrasion with silica-coated alumina particles at 2.5 bar pressure was more effective than 1.5 bar and provided more favorable 3D surface roughness parameters for micromechanical retention and adsorption mechanisms of adhesives compared to alumina particles.