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Effects of sandblasting distance and angles on resin cement bonding to zirconia and titanium

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ABSTRACT

Objectives: The aim of this study was to evaluate effects of sandblasting distance and angles resin to zirconia and titanium bonding.

Methods: Densely sintered zirconia and cp2 titanium specimens were prepared and randomly divided into groups, and then sandblasted with various distance (5 mm, 10 mm and 15 mm) and angles (45°, 60°, 75° and 90°). After surface treatment, each specimen surface underwent a silane primer application (RelyX, 3M ESPE), followed by bonding of a resin cement (RelyX Unicem Aplicap, 3M ESPE). Then, each cylindrical resin stub (diameter 3.6 mm × 2 mm) underwent a shear adhesive (bond) strength test and surface roughness evaluation. SEM evaluation and EDX analysis were used to observe surface properties of both zirconia and titanium samples. Results were statistically analyzed using analysis of variance (ANOVA) and Turkey test ($\alpha = 0.05$).

Results: Surface roughness showed a significant difference amongst the different distances and angles for both the zirconia and titanium materials and these changes in surface roughness were evident in the SEM imaging photos. As for the adhesive strength, there was a significant difference in the adhesive strength for the titanium and zirconia with different angles. In general, 75° gives the best results although this is not significantly different from 90°. However, no significant difference was observed in changes of sandblasting distance for both materials. EDX analysis at the surface revealed elements carbon, oxygen, silicon, aluminum, and zirconia on the surface.

Conclusions: Sandblasting at various distance and angles contributes differences in surface roughness when it comes to both zirconia and titanium materials. Despite both 75° or 90° sandblasting angle could yield a sufficiently high adhesive strength for resin to titanium or zirconia bonding, sandblasting at 75° seems to be optimal to increase the adhesive strength.

1. Introduction

Titanium and also in a growingly extent zirconia are the two most commonly preferred materials for dental subgingival implants. These materials fulfill the safety and biomechanical standards that have been used and suggested by researchers and dentists to be the most ideal for dental implants and other indirect dental restorations [1,2]. Both these materials have superior strength that can endure the everyday occlusal forces that teeth may undergo and they also have excellent biocompatibility without any adverse side effects [3].

Zirconia is one of the most commonly studied current ceramic in dentistry. Its ability to take different forms at different temperatures makes the material very special and unique to other materials. The most desired characteristic of zirconia is its translucent color and esthetic appeal [4]. Furthermore, the high biocompatibility and osseointegration ability enrich the usage of zirconia [5]. In fact, researchers have found that zirconia possesses similar mechanical properties to stainless steel. Some other applications for zirconia in dentistry include implant screws, abutments, bridges and crowns [6].

Titanium, on the other hand, has been the material of choice used for dental implants within the past several decades. Although titanium and its alloys are known for their biocompatibility, low

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density, and strength, their greatest characteristic is ability to osseointegrate with living bone and other tissues. Thus, titanium in dentistry is ideal for some applications with high biocompatibility and strength [2].

One of the major applications for sandblasting in dentistry, in addition to cleanse surfaces, is to increase the surface roughness. Surface roughness focuses on the topography of a surface [7]. Even with a naked eye, a surface can appear to be smooth, however with a surface roughness tester at a microlevel, we can see that a smooth surface is in fact, not really smooth. It is not surprising that with a rougher surface area, sandblasting dramatically increases the surface area.

With this is mind, sandblasting a surface can help increase surface area and result in increased micromechanical adhesion by interlocking [8]. For titanium implants, researchers have suggested that an increased surface area can result in an increase of osseointegration [9]. As for sandblasting on zirconia surfaces, researchers have argued that by sandblasting at a close distance, the mechanical properties of zirconia in fact, decrease, as it can initiate micro cracks throughout its process [3]. Under examination of a scanning electron microscope (SEM), the roughness of a surface is evident and in fact, alters the material's mechanical properties [10]. Indeed, besides sandblasting, many attempts has been done on altering the zirconia surface to improve the resin–zirconia bonding, such as laser [1], hydrofluoric acid etching [11,12], selective infiltration etching (SIE) [13,14], glazing [11], as well as chemical modification using *e.g.* silanes [15], phosphates [16] and zirconate [17]. All of these surface treatment methods seemed to give quite good results, but controversial arguments exist [18] due to non-standardized test method and the environmental difference between laboratories. Therefore, even for the most common sandblasting method, despite the theories of the optimal sandblasting distances and angles exist, there is no clear standard to ensure that the optimum bond strength is applied in particular to zirconia and titanium materials.

The adhesive strength of an interface between two materials is one of the most important characteristics it can hold. Without it, the material bonding will have no future, especially in the challenging oral conditions. In dentistry, the strength of adhesion could be evaluated in laboratory under shear or tensile modes [19]. In particular, shear mode focuses on the stress of layers of atoms or molecules displacing from one layer to the next, and tensile mode differs from shear in a way that tensile stress varies depending on the given load [20]. The shear adhesive strength (previously so-called as 'shear bond strength') measured between the resin cement and either zirconia or titanium material has been well studied and reported [19]. Therefore, studying the shear adhesive strength on zirconia and titanium with resins could be regarded as a generalized method, whereas the effects of different geometric factors of sandblasting can be evaluated.

The purpose of this laboratory study was to evaluate the effects of geometric factors, *i.e.* the distance and angles of sandblasting to find out the optimal adhesion between zirconia and titanium materials with resin cement using as an adhesion promoter, a silane coupling agent [7]. The objectives included testing whether or not there was a significant difference on the surface roughness of sandblasted titanium and sandblasted zirconia, whether or not there is a significant difference on the shear adhesive strength at different angled sandblasting, and lastly, whether or not there is a significant difference on the shear adhesive strength at different distances of sandblasting. The hypotheses were: (1) there is a significant difference in the surface roughness on either zirconia or titanium, when comparing before and after sandblasting, (2) the optimum angle for sandblasting for both materials would be 90° and a distance of 10 mm, and (3) shear adhesive strength will increase as the surface roughness of the material increases.

2. Materials and methods

2.1. Preparation of zirconia and titanium specimens

Five blocks of zirconia with the approximate size of 25 mm × 44 mm × 6 mm were obtained from Aidite (Qinhuangdao Aidite High-Technical Ceramics, China). Each block was cut into seven equal planar slices and each block was cut in half again. These blocks were then eventually cut into pieces with a height of 6.0 mm and a length and width of 13.0 mm × 16.1 mm with the use of precision saw (Micro Slice machine, Cambridge, UK). After preparing the zirconia specimens into individual slices, each sample was individually wet-polished on the manual polisher (Lunn Major, Struers, Denmark) using a series of silicon carbide abrasive paper. Each piece was polished on the 500-grit abrasive paper under running water for 30 s, followed by the 1000-grit abrasive paper for additional 30 s. Following polishing the zirconia samples was sintered at the temperature of 1500 °C. After sintering zirconia the specimen sizes shrunk approximately 57.1% in volume. For titanium, pre-cut planar specimens of 150 mm × 30 mm × 1 mm were obtained.

2.2. Sandblasting treatment

A sandblasting machine (Shofu Pen-Blaster™, Shofu Dental MFC, Kyoto, Japan) with a silica-coated alumina powder, with a particle size of 110 µm (Rocatec™ Pre, 3M ESPE, St. Paul, MN, USA) were used. The sandblasting pen (*i.e.* wand) with the tip nozzle size 3 mm was used in rotational movements, and the operational pressure was constant (3.5 bar) for 15 s for a 1.0 cm^2 substrate area. These settings were used throughout the study. A custom-made device was used in order to ensure the consistency of distance and angle change. The design of the device is shown in Fig. 1. This device allowed for the sandblasting pen to hold in place at the desired angles (45°, 60°, 75°, and 90°). In addition, custom-made spacing blocks with different height were used in order to measure the desirable specimen distances (5.0 mm, 10.0 mm and 15.0 mm) from the sand-blasting pen.

Following sand-blasting, each specimen was washed and rinsed with 70% ethanol (BDH Reagents & Chemicals, Poole, UK), then rinsed with deionized water (Milli-Q, Millipore, MA, USA), and left to dry at room temperature overnight. Once the specimens were

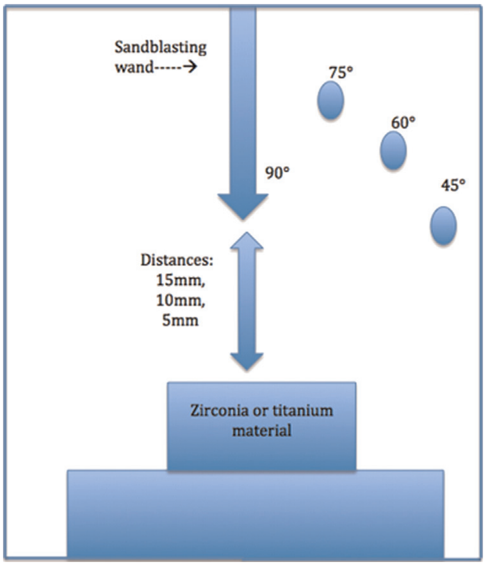


Fig. 1. Design of the device created to emulate the different sandblasting angles and distance.

dried, a silane primer, (3M RelyX™ ESPE, Seefeld, Germany) was applied to the spots where they were sandblasted. Then, these specimens were left to dry at room temperature overnight again.

2.3. Bonding procedure

The specimens were allowed to dry overnight and silanized, then resin cement (RelyX™ Unicem Aplicap™, 3M ESPE, Seefeld, Germany) stubs of 2 mm in height were individually applied to each specimen using a polyethylene mold of roughly ~3.6 mm in diameter and light-cured from the top for 40 s by a halogen light source (Elipar™ 2500, 3M ESPE, Seefeld, Germany), according to the manufacturer's instructions. Each piece of zirconia allowed for two stubs on each surface, while the titanium places allowed for eight stubs per sheet. After light-curing, the mold was carefully removed around the stub by pressing firmly the stub with a hand instrument.

2.4. Surface roughness test

Prior to sandblasting, each specimen was to undergo a surface roughness test. The surface roughness test was conducted using an electro-mechanical profilometer (Surtronic 3+, Taylor Hobson, Leicester, England). An average calculation of the surface roughness (R_a) was used for the statistical analysis to describe eventual general trends. After the sandblasting procedure, each specimen was cleaned with the 70% ethanol, and then pat dried in order to wash off any remaining powder particles. Once the specimens were dried, the same procedure of surface roughness testing was conducted to test the surface roughness difference before and after surface treatment.

2.5. Shear adhesive strength test

Following the resin cement application, shear adhesive strength test was conducted by using the universal testing machine (ElectroPuls™ E3000, Instron Industrial Products, Grove City, PA, USA), at a cross head speed of 1 mm/min and a maximum force of 500 N/mm^2. The zirconia and titanium samples were mounted in different custom made jigs to test the adhesive strength. Zirconia samples were placed in a slot and held in place by a screw. On the other hand, the titanium sheets were guided through a small gap and held in place with two screws.

2.6. Scanning electron microscopy

Each sample was examined under the scanning electron microscopy (SEM, Hitachi S-3400N VP-SEM, High Technologies Europe, Krefeld, Germany). This machine allowed a better understanding of the surfaces from the samples and also analysis of fracture morphology. The titanium specimens were fixed onto the platform in the machine directly without any prior treatment. However, due to the poor conductivity of zirconia, the zirconia samples were glued onto aluminum specimen holder stubs and coated with gold in an ion sputterer (JFC-1100, JEOL, Tokyo, Japan).

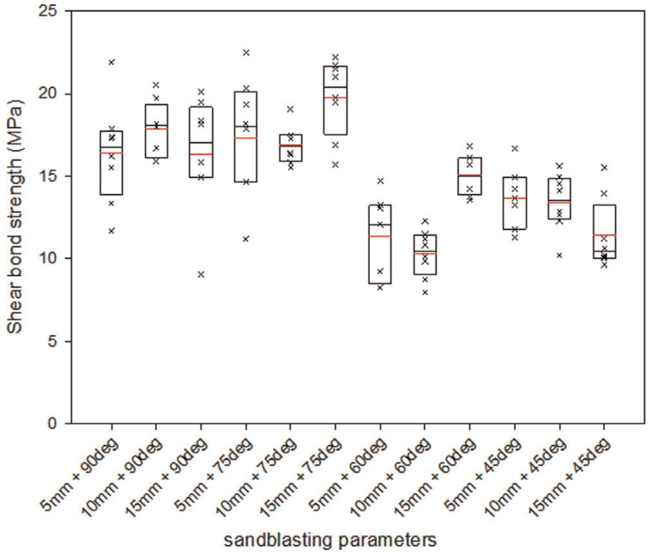


Fig. 2. Box-plot of SBS of the titanium test groups ($n=8$). The black line denotes the median, red line denotes the mean, a box denotes a 50% quartile, and black × denotes the data. 'mm' denotes the nozzle distance; 'deg' denotes the angle of the sandblasting pen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

2.7. Surface chemical composition

Surface chemical composition was evaluated by using a scanning electron microscope (Hitachi S-3400N VP-SEM, Hitachi, High Technologies Europe, Krefeld, Germany) which entailed an electron dispersive X-ray spectroscopy (EDX) module, which allowed for detection of elemental mapping (INCAx-sight EDS Detectors with INCA Energy Software, Oxford Instruments, Oxfordshire, UK). The EDX detector used a super atmospheric thin window, a 20 kV accelerating voltage with a 40 s acquisition time. Analysis of the percent weight concentration was averaged over the selected region of the debonded surface.

2.8. Statistical analysis

The mean shear adhesive strength of each group was analyzed using a two-way ANOVA with the shear adhesive strength as the dependent variable and the degrees and distances as the independent variable (SPSS Statistics 20; IBM). The surface roughness analysis for titanium samples and zirconia samples was also analyzed. α at 0.05 was used as the standard to establish significance.

Table 1
Mean R_a (SD, μm) for titanium (Ti) and zirconia (ZrO_2) samples.

Distance (mm)	Degree								Control (without sandblasting)	
	45°		60°		75°		90°			
	Ti	ZrO_2	Ti	ZrO_2	Ti	ZrO_2	Ti	ZrO_2	Ti	ZrO_2
5.0	1.08 (0.08)	0.49 (0.06)	1.17 (0.17)	0.49 (0.03)	0.91 (0.12)	0.55 (0.07)	0.88 (0.04)	0.81 (0.30)	0.69 (0.06)	0.38 (0.05)
10.0	0.99 (0.09)	0.53 (0.10)	1.04 (0.13)	0.53 (0.10)	0.94 (0.10)	0.69 (0.11)	1.14 (0.30)	0.53 (0.07)		
15.0	0.98 (0.03)	0.51 (0.05)	0.98 (0.03)	0.74 (0.12)	0.95 (0.03)	0.58 (0.09)	0.95 (0.10)	0.53 (0.01)		

3. Results

3.1. Surface roughness

The surface roughness (R_a) of zirconia and titanium were examined, to see if there was a significant difference to the surface roughness before and after sandblasting treatment (Table 1). All the sandblasted specimens have shown a higher roughness than the control. As for titanium samples, the highest recorded average of surface roughness was at 5.0 mm and 60° with an average of 1.17 µm, while the lowest surface roughness was 5.0 mm and 90° with an average of 0.88 µm. When comparing within the distance groups, at 10.0 mm, the highest surface roughness had an average of 1.14 µm at 90° and the lowest surface roughness had an average of 0.94 µm at 75°. At 15.0 mm, the highest average was 0.98 µm for both 45° and 60°, while the lowest average was at 0.95 µm for both 75° and 90°.

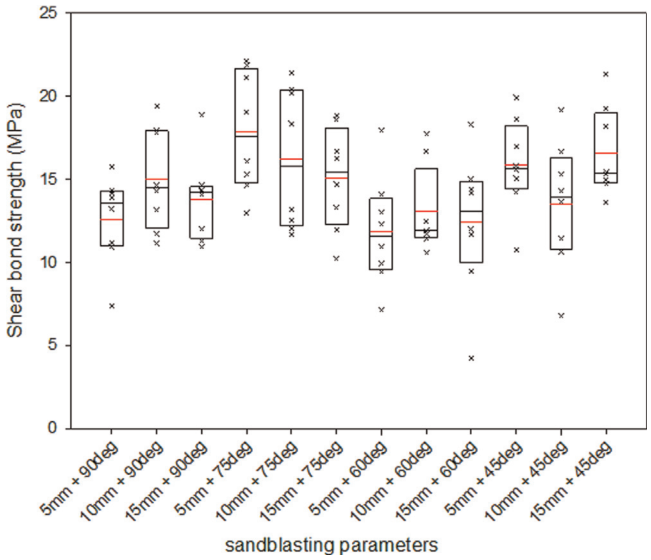


Fig. 3. Box-plot of SBS of the zirconia test groups ($n=8$). The black line denotes the median, red line denotes the mean, a box denotes a 50% quartile, and black × denotes the data. 'mm' denotes the nozzle distance; 'deg' denotes the angle of the sandblasting pen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

For zirconia samples, the highest surface roughness was at 5.0 mm and 90° at 0.81 µm and the lowest surface roughness being 5.0 mm at 45° and 60° at 0.49 µm. When comparing within the distance groups, at 10.0 mm, the average surface roughness was the same for 45°, 60°, and 90° at 0.53 µm. However, at 75°, there was a slightly higher average at 0.69 µm. For 15.0 mm, the highest surface roughness was at 60°, while the lowest surface roughness was at 45°, although the averages for 60° and 90° were quite similar.

3.2. Shear adhesive strength

Both zirconia and titanium samples were tested and there was a significant difference in the adhesive strength to various angles and distances of sandblasting. The box plots diagrams in Figs. 2 and 3 represent the average means in relation to the other groups divided into categories of the distances and angle differences for titanium samples and zirconia samples, respectively.

For titanium samples, a two-way ANOVA test was conducted on these samples. The two-way ANOVA test revealed that there was no significant difference amongst distances. However, for 'degree'

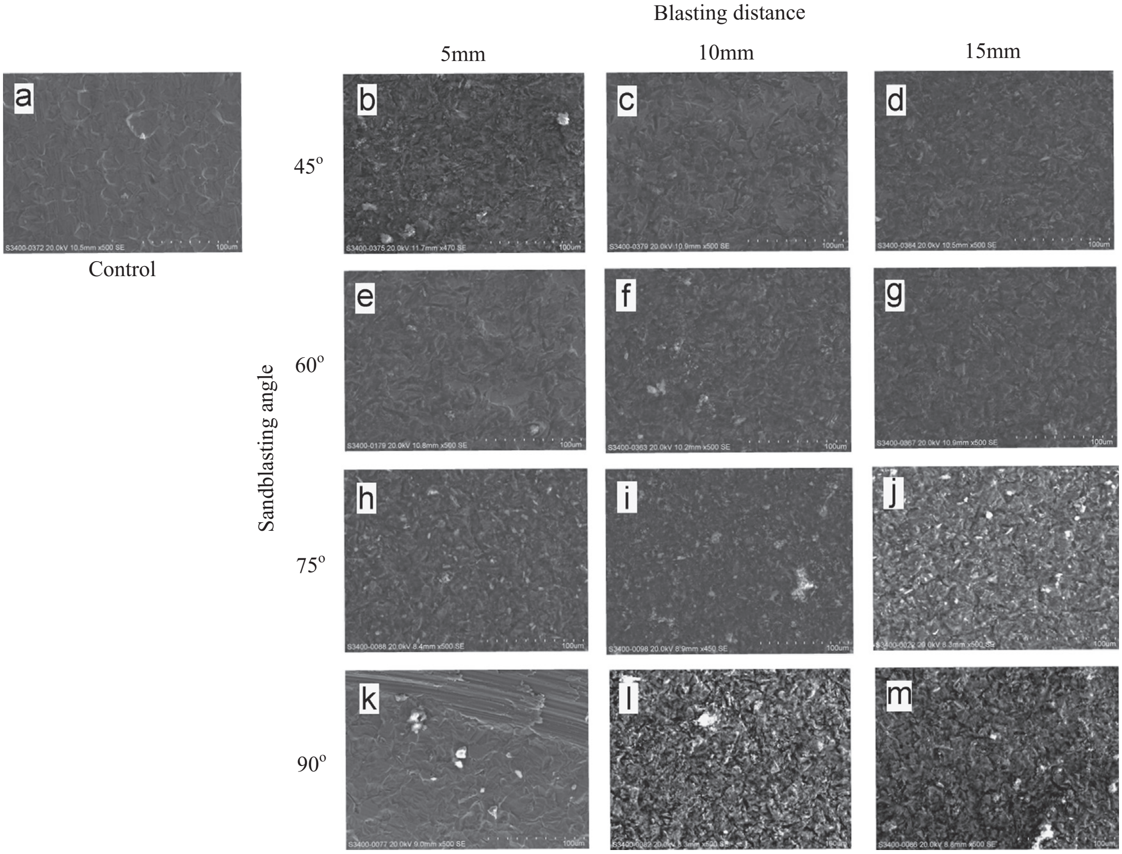


Fig. 4. Representative SEM for debonded titanium surfaces. (a) Control, no sandblasting treatment, (b–d) 45°, at 5 mm, 10 mm and 15 mm, (e–g) 60°, at 5 mm, 10 mm and 15 mm, (h–j) 75°, at 5 mm, 10 mm and 15 mm and (k–m) 90°, at 5 mm, 10 mm and 15 mm.

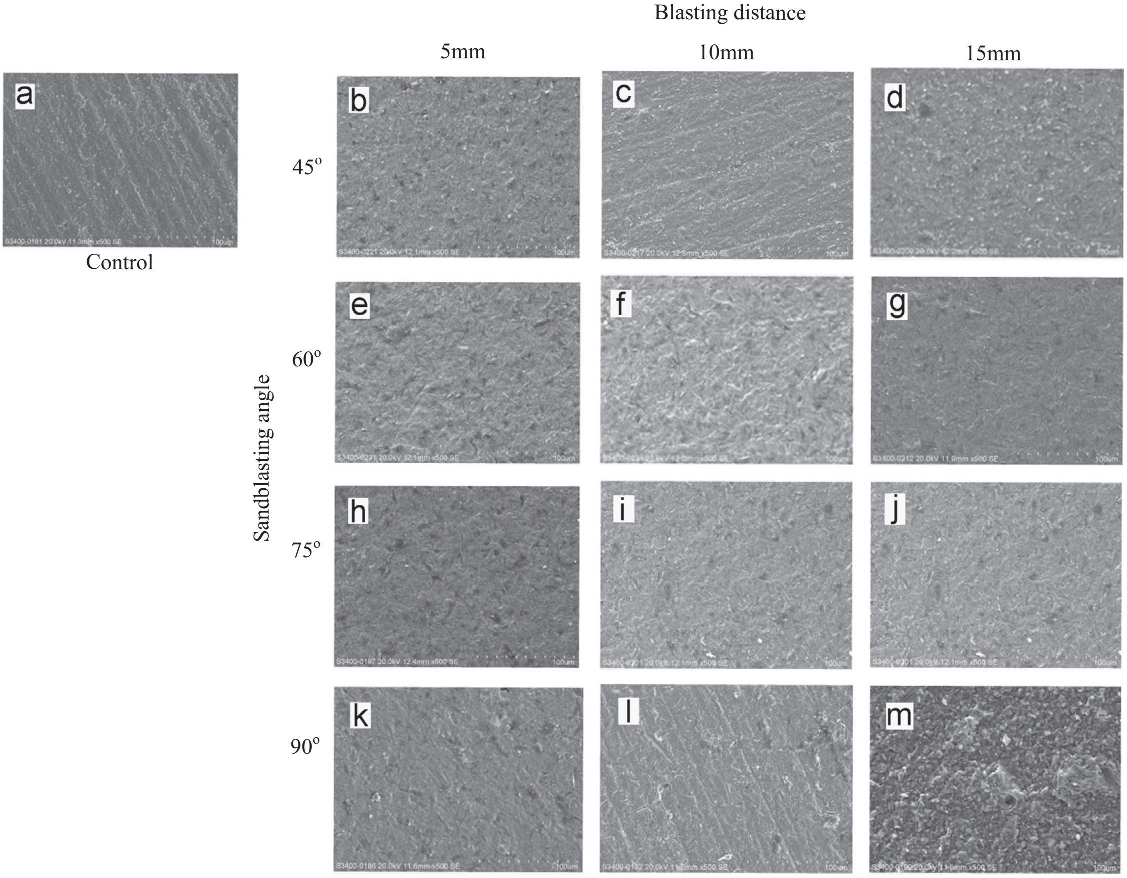


Fig. 5. Representative SEM for debonded zirconia surfaces. (a) Control, no sandblasting treatment, (b–d) 45°, at 5 mm, 10 mm and 15 mm, (e–g) 60°, at 5 mm, 10 mm and 15 mm, (h–j) 75°, at 5 mm, 10 mm and 15 mm and (k–m) 90°, at 5 mm, 10 mm and 15 mm.

or ‘distance*degree’, there was a significant difference in both with $p<0.001$. When conducting a *posthoc* Tukey HSD test, 45° had no significant statistical difference to 60° with $p=0.731$, nor did 75° to 90° with $p=0.858$. However, other pairwise comparisons had a significant statistical difference with $p<0.001$. An overall ranking of the degree differences was $75°>90°>45°>60°$. As for zirconia samples on the other hand, a two-way ANOVA test was conducted since the shear adhesive strength data had equal variances. Shear adhesive strength in distances had a ranking of 15 mm > 5 mm > 10 mm, however, there were no statistically significant differences in the shear adhesive strength in the groups ‘distances’ and ‘distance*degree’. However, amongst the degree variations, there was a statistically significant difference with a *p*-value of 0.001. A *post hoc* Tukey HSD test was conducted and revealed that 75° had no significant difference to 45° and 90°, but a significant difference to 60° ($p<0.001$). In addition, 45° had no significant difference to 75° and 90°, yet, had a significant difference to 60° ($p=0.015$). Lastly, 90° had no statistical significance to three other degree variations. An overall ranking of the degree differences was $75°>45°>90°>60°$.

3.3. Scanning electron microscopy

The SEM images of the sandblasted surfaces of specimens after shear adhesive strength testing are shown in Figs. 4 and 5. Each image has a control to compare the change in surface morphology. These images were taken at magnifications of 450× to 500× to get a clearer image.

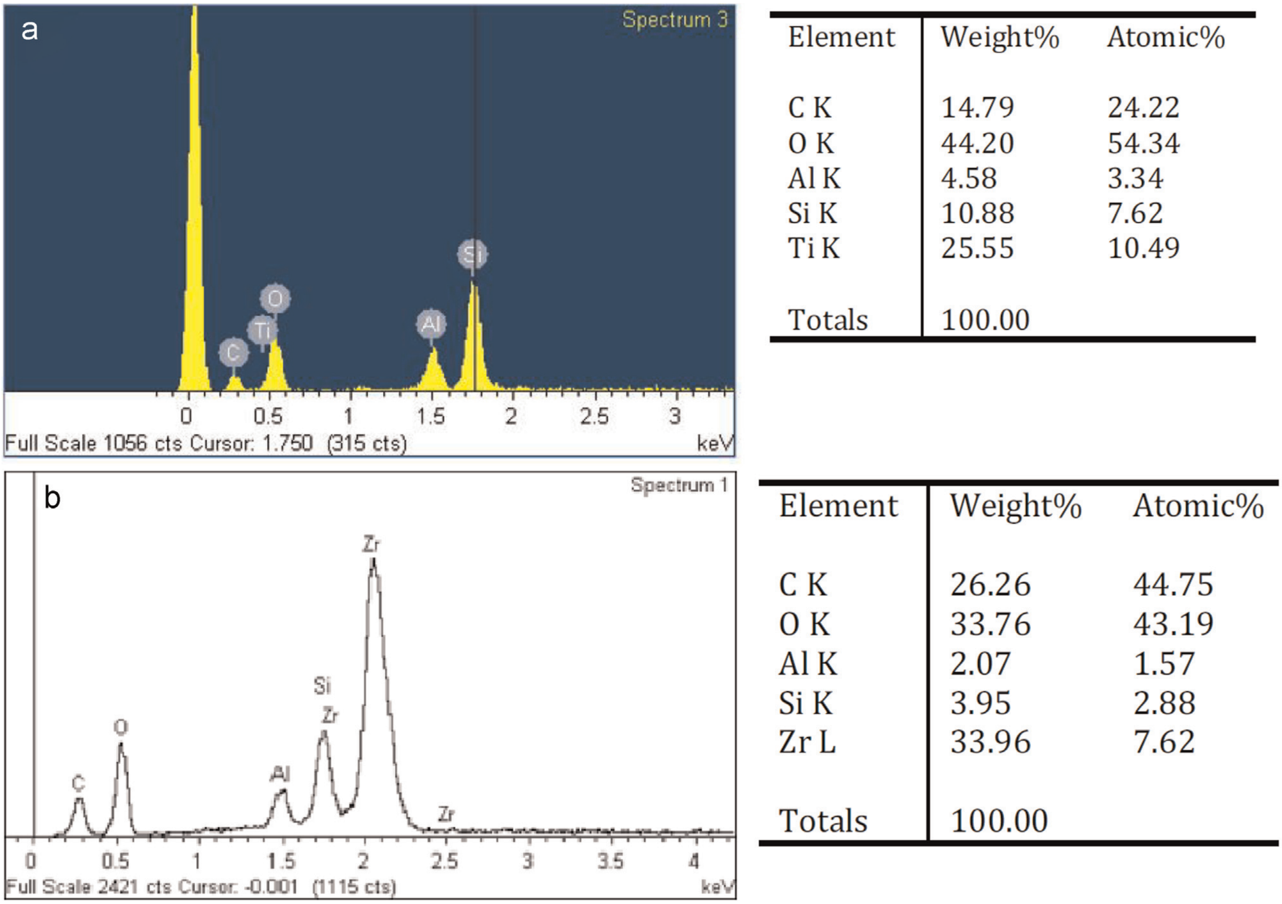
3.4. Surface chemical composition

Fig. 6 shows the representative EDX analysis of debonded surface of (a) titanium and (b) zirconia. Based on the EDX analysis, the majority of elemental content for titanium sample surfaces after sandblasting and resin cement bonding were carbon, oxygen, silicon, aluminum and titanium. As for zirconia sample surfaces, the majority of elemental content were carbon, oxygen, aluminium, silicon and zirconium.

4. Discussion

Sandblasting is a common procedure used in dentistry to roughening and remove impurities from indirect restoration surfaces. Whether it is performed at the dentist's chair-side or in a laboratory, the simple procedure of sandblasting a material can be significant. Although this procedure is used fairly common in the field of dentistry, the standard procedure that was rely only on the basic science theory (*i.e.* kinetic energy$=\frac{1}{2}mv^2$, where m is mass and v is velocity). This might not be able to ensure that sandblasting could accomplish its task of enhancing micromechanical retention. Thus, this study was conducted in order to discover the optimum distance and angle for sandblasting in order to ensure the greatest strength for bonding two materials together. The results might give more understanding as no other similar studies have been reported previously in dentistry.

As a result to sandblasting, the surface roughness of a material increases regardless of the type of material it is. The surface roughness was examined amongst zirconia and titanium samples, and there was very little variation amongst the different angles,



Element	Weight%	Atomic%
C K	14.79	24.22
O K	44.20	54.34
Al K	4.58	3.34
Si K	10.88	7.62
Ti K	25.55	10.49
Totals	100.00	

Element	Weight%	Atomic%
C K	26.26	44.75
O K	33.76	43.19
Al K	2.07	1.57
Si K	3.95	2.88
Zr L	33.96	7.62
Totals	100.00	

Fig. 6. EDX analysis of (a) titanium and (b) zirconia on representative sample after adhesive strength test at the debonding surface.

but amongst the different distances, there was some difference. For zirconia samples, the greatest surface roughness was produced when the sand-blasting pen was at 5 mm away from the sample at a 90° angle. An explanation for this might be because the pen focuses adjacent and very close to the surface. The lowest surface roughness was also at 5 mm but at 45° and 60°. A reason for this might be because at such a close distance (but a wider angle) the powder particles do not have as much kinetic energy and is easily dispersed when hitting the surface. As for titanium samples, the highest surface roughness was at 60° and 5 mm, while the lowest was at 90° and 5 mm. Based on these results, titanium and zirconia appear to have the opposite results with surface roughness. A possible explanation for these results might be that the powder particles were hitting and embedding into the titanium surface with the uneven, thin titanium oxides [21].

These oxide films are a product of spontaneous reaction with air and they might scatter and absorb the force (and the kinetic energy) that will eventually penetrate to the higher strength bulk metal. Thus, at 90° the force is less than expected and therefore remove less surface. Unlike the zirconia in which the surface and the bulk are essentially the same crystalline structure, there is no scattering of force, at least in principle. Thus, general consensus of roughness creation by kinetic energy withholds. Nevertheless, further investigations are necessary to clarify the situation.

Shear adhesive strength tests were conducted in order to evaluate optimum distance and angles for sand-blasting for zirconia and titanium samples. The results in this study for the shear adhesive strength varied amongst different distances and angles and no common trend was identified. For zirconia samples, the overall ranking was 75° > 45° > 90° > 60°, whilst for titanium samples, 75° > 90° > 45° > 60° was the overall ranking. Interestingly, 60° perform the worst and 75° perform the best in both materials. The 60° produced contradictory surface roughness on both materials. It seems to be the shear adhesive strength in the test adhesive system that did not follow the general thought that a higher the surface roughness would increase the micromechanical retention, which should directly increase the shear adhesive strength. This substantiates the silane coupling agent [8], the adhesive, possibly played a very important role *i.e.* providing the chemical bonding which might significantly influence the shear adhesive strength over the micromechanical interlocking [22].

For both titanium and zirconia, the 90° and 75° sandblasting angles with various distances 5–15 mm gave no statistically significant difference on the shear adhesive strength. Indeed, in the real situation, the ideal 90° condition which maximizes the kinetic energy is not always obtainable. There are always conditions, such as variability of the sandblasting pen location and the handling of materials by operators. These would influence the sandblasting quality which would then affect the cementation quality and durability. Thus, despite the limitation of this study, we could say both angles used for sand-blasting provide a sufficiently high strength in the cementation of these two materials using the existing adhesive system.

Comparing two very different materials (a ceramic and a metal) may be a reason for this inconsistency. In the case of zirconia, although the manufacturer claimed this zirconia is compatible with another brand of zirconia and could be sintered with the same condition, in another recent study [23], it was found that this zirconia could not be fully sintered. Thus, internal flaws appeared in the sample and thus induced the materials would not be the claimed strong materials. Therefore, the slightly soft zirconia became slightly flexible under the jig mounting, and possibly chipped-off the adhered resin cement. Due to the small sample size for both materials, it limited the number of analyses that could be performed and the results of the current tests. In our future studies, a greater number of sample sizes would be ideal in order to ensure that none of the sample results are a coincidence.

For titanium samples, there was a high signal for silicon and aluminum. This could have been remnants of the powder particles used from sandblasting. As for the zirconia samples, there were (not surprisingly) high signals for Zr and O. This reveals that the sample's composition of ZrO_2 was detected more than the remnants from the sand particles or the resin cement. The lowest elemental signals observed were silicon and aluminum, signifying that there were very little remaining sandblasting particles on the surface. In summary, further investigation in this area can focus on different particle sizes, different types of sand particles, and or different pressures of sandblasting.

In general, the hypothesis (1) is accepted since there is a significant difference in the surface roughness before and after sandblasting on either zirconia or titanium. Hypotheses (2) and (3) are rejected since the optimum sandblasting angle for resin adhesion to both materials would be 75° according to this test condition, and the distance (5–15 mm) and roughness have no significant effect on shear adhesive strength.

5. Conclusion

Sandblasting at different distances and angles contributes differences in surface roughness when it comes to both zirconia and titanium materials. However, when it comes to the adhesive strength, there is a significant difference in strength for both titanium and zirconia materials at varying degrees but not varying the distance. Despite both 75° or 90° sandblasting angle could yield a sufficiently high adhesive strength for titanium material and zirconia material, sandblasting at 75° seems to be optimal to increase the adhesive strength.

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