THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Influence of additive and subtractive zirconia and lithium disilicate manufacturing on tensile bond strength and surface topography

Andreas Kessler, DMD, MSc, PhD,[ab] Thilo Reichert, DMD, PhD,[c] Stefanie Lindner, DMD, MSc, PhD,[d] Anja Liebermann, DMD, MSc, PhD,[e] Moataz El Gezawi, BDS, MSc, PhD,[f] and Dalia Kaisarly, BDS, MSc, PhD[g]

ABSTRACT

Statement of problem. Although bonding is important for long-term clinical success, studies on the bonding of additively manufactured ceramics are sparse.

Purpose. The purpose of this in vitro study was to determine the influence of manufacturing methods, additive (LCM) versus subtractive (CAM). and ceramic materials, zirconia (ZrO_2) and lithium disilicate (LiSi), on the tensile bond strength (TBS), failure mode, and surface roughness of ceramics.

Material and methods. A total of 240 ceramic specimens (n=60/group; 2×2×10 mm) were prepared. Two additively manufactured (LCM-printed) ceramics, LiSi and ZrO_2 (Lithoz), subtractively manufactured LiSi (IPS e.max CAD), and subtractively manufactured ZrO_2 (KATANA Zirconia HTML PLUS) were evaluated. From each material, 40 specimens were bonded together (n=20 ceramic-ceramic specimens/group), and 20 specimens were bonded to equally sized human dentin specimens (n=20 ceramic-dentin specimens/group). The ZrO_2 specimens were airborne-particle abraded (Al_2O_3, 50 μm, 0.1 MPa), and the LiSi specimens were etched with hydrofluoric acid. Then, a universal primer (Monobond Plus) was applied. After the dentin was coated with an etch-and-rinse adhesive (Syntac Classic), the specimens were bonded with luting composite resin (Variolink Esthetic DC), light polymerized for 40 seconds, thermally aged (10 000 cycles between 5 °C and 55 °C), tested for TBS, and statistically analyzed (1- and 3-way ANOVA and Weibull analysis). The ceramic surface was examined with scanning electron microscopy, and surface roughness was measured with digital microscopy before and after surface pretreatment.

Results. TBS varied between 5.88 ±2.22 MPa and 6.34 ±2.26 MPa in the ceramic-dentin groups and 12.40 ±1.56 MPa and 18.82 ±5.92 MPa in the ceramic-ceramic groups. No significant difference was observed regarding the manufacturing method and material for different bonding conditions ($P>.05$). Additive and subtractive LiSi showed the highest reliability with m=18.27. The ceramic-ceramic specimens failed cohesively in the luting composite resin, whereas the ceramic-dentin specimens failed adhesively.

Conclusions. The manufacturing method and material used had little effect on bond strength values or surface properties. The recently introduced printed materials performed similarly to conventionally milled materials. (J Prosthet Dent 2024;132:623.e1-e7)

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.
Additive material specimens provided by Lithoz.
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
[a]Assistant Professor, Department of Prosthetic Dentistry, Faculty of Medicine, Center for Dental Medicine, Medical Center - University of Freiburg, University of Freiburg, Freiburg, Germany.
[b]Assistant Professor, Department of Conservative Dentistry and Periodontology, University Hospital, Ludwig-Maximilian-University of Munich, Munich, Germany.
[c]Postdoctorate, Department of Conservative Dentistry and Periodontology, University Hospital, Ludwig-Maximilian-University of Munich, Munich, Germany.
[d]Assistant Professor, Department of Conservative Dentistry and Periodontology, University Hospital, Ludwig-Maximilian-University of Munich, Munich, Germany.
[e]Full Professor and Department Head, Department of Prosthetic Dentistry, University Hospital of Cologne, Cologne, Germany.
[f]Full Professor, Department of Restorative Dental Sciences, Imam Abdulrahman Bin Faisal University, Dammam, Saudi Arabia.
[g]Assistant Professor, Department of Conservative Dentistry and Periodontology, University Hospital, Ludwig-Maximilian-University of Munich, Munich, Germany.

Clinical Implications

Based on this investigation, additively manufactured or 3D printed ceramics have bond strength values comparable with those of subtractively manufactured ceramics.

Subtractive computer-aided design and computer-aided manufacturing (CAD-CAM) has become a widespread and successful dental manufacturing method.[1] However, subtractive manufacturing has disadvantages, including the high raw material waste, necessary correction of the milling bur, access of milling tools, and occurrence of wear.[2] Microscopic surface defects, including cracks, may occur and can weaken the restoration.[3]

Additive manufacturing or 3-dimensional (3D) printing could address these disadvantages[4] and is already an established process in the production of metals and polymers.[5–7] However, the additive manufacture of ceramic restorations is still limited because of the challenging manufacturing environment of high temperatures and a corrosive atmosphere.[8]

The first attempts to print ceramics were described in 1990 by Marcus et al[9] and Sachs et al.[10] Currently, lithography-based methods, including stereolithography (SLA) and digital light- processing (DLP), can be used to print green body ceramic by selectively polymerizing the slurry, which contains ceramic powder and a liquid photosensitive binder.[11–13] The formed objects are then thermally debinded and finally sintered to achieve dense ceramic parts. The method has been evaluated using ZrO_2, alumina, tricalcium phosphate, and lithium disilicate ceramics.[14] Through additive manufacturing, material costs can be reduced, complex structures can be constructed, and material and esthetic properties can be selectively or gradually modified.[2] The additive manufacturing of dental ceramic is of high interest, as the materials exhibit promising mechanical properties.[15,16]

In addition to mechanical proprieties, the bond between the ceramic and tooth structure is among the crucial points that affect the longevity of a restoration.[17–19] However, information regarding the influence of the fabrication method (additive versus subtractive) on the surface roughness and bond strength of ceramics to substrate dentin or ceramics is lacking. Therefore, this study aimed to evaluate the influence of the manufacturing method (subtractive versus additive) and material (zirconia and lithium disilicate) on the tensile bond strength (TBS),[20–26] fracture mode,[27–29] and surface roughness.[30–33] The null hypotheses were that ceramic-ceramic and ceramic-dentin bond strength and surface topography of ZrO_2 and LiSi would not differ between additively and subtractively manufactured ceramics.

MATERIAL AND METHODS

Two additively manufactured ceramics, lithium disilicate (LiSi) and zirconia (ZrO_2) (Lithoz), subtractively manufactured LiSi IPS e.max CAD blocks (Ivoclar AG), and subtractively manufactured ZrO_2 KATANA Zirconia HTML PLUS (Kuraray Noritake) were investigated. A total of 60 specimens (2×2×10 mm) were prepared from each material, and the tensile bond strength (TBS) of both ceramic-to-ceramic and ceramic-to-dentin was determined. Moreover, the surface was analyzed by using scanning electron microscopy and surface roughness measurements.

The additive ceramic specimens were fabricated using a 3D printer (CeraFab S65 Medical; Lithoz) based on lithography-based ceramic manufacturing (LCM) technology. An experimental slurry with a 45 vol% ceramic filler content was developed to manufacture LiSi. For the ZrO_2, a commercially available slurry (LithaCon 3Y 210; Lithoz) with a 48 vol% filler content was used. The printing process involved 540 layers, each 25 μm thick and lasted a total of 5.4 hours for each ceramic. The ceramic specimens were cleaned with compressed air, and excess unpolymerized material was removed with a cleaning fluid (LithaSol 20; Lithoz).

Debinding the LiSi specimens was done by heating them in a furnace (L40; Nabertherm GmbH) to 430 °C, followed by a dwell time of 6.5 hours. The final sintering was performed in a ceramic furnace (Programat CS3; Ivoclar AG) at 900 °C with a dwell time of 1 hour. Debinding and sintering of the ZrO_2 specimen were carried out in a single run using a high-temperature furnace (Nabertherm L40; Nabertherm). The specimens were gradually heated to a sintering temperature of 1450 °C, which removed the organic photopolymer matrix through pyrolysis, and held for 2 hours.[34]

Cuboids of the LiSi ceramic specimens were milled from blocks (IPS e.max CAD; Ivoclar AG) (SiO_2, Li_2O, Al_2O_3, K_2O, ZrO_2, P_2O_5, lithium disilicate crystals 3 to 6 μm about 70 wt%) using a milling unit (inLab MC X5; Dentsply Sirona). The specimens were cut from these cuboids in the precrystallized, blue, state with a precision saw (Isomet Low Speed; Buehler) under water cooling and then crystallized at 840 °C in a furnace (Programat EP 5000; Ivoclar AG).

The ZrO_2 specimens were milled from a blank (KATANA Zirconia HTML PLUS; Kuraray Noritake) with a composition of ZrO_2 Y_2O_3, TiO_2 wt% cubic phase < 50, Grain size 0.63 in the milling unit (inLab MC X5; Dentsply Sirona) and sintered at up to 1500 °C with a dwell time of 2.25 hours in a sintering furnace (inLab Profire; Dentsply Sirona). Both the additively and subtractively manufactured ZrO_2 contained a 3-mol% yttria content.

A total of 40 freshly extracted human third molars were collected and stored in Ringer solution and 2% sodium azide. The experimental procedures had been approved by the ethical committee of the medical faculty (21–1089KB). The occlusal one-third of the dental crown was removed, and a smear layer created on the dentin surface by using 600-grit abrasive paper (Leco SS-200; LECO Instruments).[35] Subsequently, the specimens were cut from the teeth with a precision saw (Isomet Low Speed; Buehler) under water coolant. These specimens had a 2×2-mm dentin surface with a smear layer and were stored in distilled water until bonding to the ceramic specimen, within 3 hours. Tensile bond strength was tested in a split-tooth design, and the specimen of 1 tooth was divided equally between the 2 types of ceramics being compared.

For the TBS test for each ceramic type, 60 ceramic specimens and 20 dentin specimens were tested, with 20 specimens of 1 ceramic type bonded to 20 specimens of the same material and the remaining 20 specimens bonded to 20 dentin specimens. This resulted in 20 ceramic-to-ceramic specimens and 20 ceramic-to-dentin specimens for each ceramic type. The specimens were measured, examined for defects under a light microscope (BMS 74956; Breukoven) at ×200 magnification, and cleaned in an ultrasonic bath with distilled water for 5 minutes before the bonding process.

Both additively and subtractively manufactured LiSi ceramics were etched with 5% hydrofluoric acid (HF) (Vita Ceramic Etch; Vita Zahnfabrik) for 20 seconds, and a universal primer (Monobond Plus; Ivoclar AG) was applied for 60 seconds. Before primer application, the additively and subtractively manufactured ZrO_2 specimens were airborne-particle abraded with 50-μm aluminum oxide (Al_2O_3)[36,37] particles at a distance of 10 mm at 0.2 MPa for 10 seconds. The dentin specimens were etched (Total Etch; Ivoclar AG) and conditioned with an etch and rinse adhesive system (Syntac Classic; Ivoclar AG) according to the manufacturer's instructions.

For mutual bonding, a specially designed specimen support was 3D printed, which allowed the specimens to be bonded to each other with precision (Fig. 1). A luting composite resin (Variolink Esthetic LC; Ivoclar AG) was used and polymerized from 2 sides with a light intensity of 1200 W/cm^2 (Bluephase Style; Ivoclar AG) for 20 seconds on each side. The excess was removed with a polishing wheel, and the specimens were stored in distilled water at 37 °C for 24 hours. The specimens were artificially aged by thermal cycling (HaakeW15; Thermo Haake) between 5 °C and 55 °C for 10 000 cycles, with a dwell time of 30 seconds and a transfer time of 5 seconds

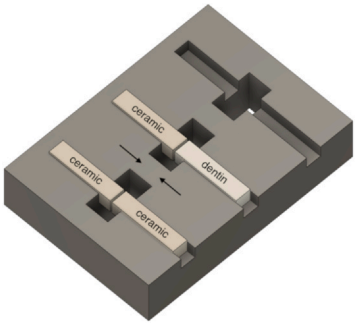

Figure 1. Specially designed 3-dimensionally printed specimen support produced to facilitate bonding of specimens to each other with precision.

TBS was determined using the tensile test system (TC 550; Syndicad Ingenieurbüro). The specimens were aligned horizontally in the tensile direction, adhesively attached to the specimen holder with a luting composite resin (Variolink Esthetic LC; Ivoclar AG), and tested at a cross-head speed of 0.5 mm/minute until fracture. The tensile strength was calculated by dividing the fracture load (N) by the bonding area (mm^2). The fracture types were analyzed under a light microscope (BMS 74956; Breukoven) at ×200 magnification, and the fracture mode identified as adhesive failure between luting composite resin and ceramic, adhesive failure between luting composite resin and dentin, cohesive failure in luting composite resin, or mixed failure.

The ceramic surfaces were analyzed after manufacturing and after surface treatment with etching in the LiSi group or airborne-particle abrasion in the ZrO_2 group. The same ceramic specimens that had been used for TBS were examined except for the subtractively manufactured ceramics which were milled from IPS e.max CAD blocks using a milling and grinding unit (MC X5; Dentsply Sirona) and subsequently crystallized in a furnace (Programat EP 5000; Ivoclar AG). The surfaces of the selected test specimens were conditioned as for TBS according to the manufacturer's instructions.

Eight ceramic specimens (n=1) were cleaned in an ultrasonic bath for 5 minutes, air dried for 24 hours, gold sputter-coated (B7391 Target 80% Au, 20% Pd; Plano), and then examined with a scanning electron microscope (ZEISS GEMINI FESEM, SUPRA 55 VP; Carl Zeiss SMT AG) at ×1000 and ×4000 magnifications.

The surface roughness of the treated and untreated ceramic was measured with a digital microscope, and its corresponding zoom objective (microscope VHX-970FN, VH-ZST; KEYENCE) at × 2000 magnification. For each ceramic type and treatment, a total of 4 specimens were examined, on which 6 measurements were made to yield n=24 measurements of surface roughness (Sa).

The data of the TBS test and the surface roughness Sa were tested for normal distribution by using the Shapiro–Wilk test. The data of the TBS test were subjected to a 3-way ANOVA to investigate the influence of the manufacturing, the ceramic, and the bonding substrate parameters. The reliability of the adhesive bond was analyzed by using Weibull statistics.[7,38,39] Sa was statistically analyzed using 1-way ANOVA and the

Tukey post hoc test with a statistical software package (IBM SPSS Statistics, v28.0; IBM Corp) (α=.05).

RESULTS

The mean and standard deviation values (MPa) for TBS are summarized in Table 1 for the different materials, bonding conditions, and manufacturing groups; the 3-way ANOVA determined no significant 3-way interaction, F(1, 152)=0.260, R-squared 0.652. The interaction was not significant between manufacturing and ceramic (*P*=.937) or manufacturing and bonding substrate (*P*=.087) but was significant between ceramic and bonding substrate (*P*<.001). The analysis of the fracture types identified 2 groups. Specimens bonded to the corresponding ceramic fractured cohesively in the luting composite resin. Specimens bonded to dentin showed adhesive failure between the luting composite resin and dentin. The highest values for the Weibull modulus were achieved by the ZrO_2 bonded to ZrO_2 groups, with 9.61 for the additive and 5.07 for the subtractive group (Table 2, Fig. 2).

The scanning electron microscope images are displayed in Figure 3. The additive manufactured LiSi showed a homogenous surface, and the subtractive manufactured LiSi appeared uneven with grinding debris. In the additive manufactured LiSi, etching with HF revealed more rod-shaped lithium disilicate crystals, which appeared angular and pointed compared with the rounded plate-shaped crystals of the subtractively manufactured LiSi. The rod-shaped crystals (approximately 2.5 μm) were larger than the large plate-shaped crystals (approximately 1.5 μm) of the subtractive LiSi. On the surface, the average grain size appeared higher in the untreated subtractive manufactured ZrO_2 than in the additive manufactured ZrO_2. After airborne-particle abrasion, no grains were observed, and any differences between the 2 ZrO_2 specimens diminished.

The 1-way ANOVA revealed a significant difference among the groups (*P*<.001): the roughness values (Sa) of the 2 LiSi groups were significantly different (*P*<.001) and higher than those of the ZrO_2 groups (Table 3). The roughness of the additive LiSi was statistically similar (*P*=.984) after etching with HF (3.70 ±0.60 μm and 3.90 ±0.28 μm). The values of the subtractively manufactured LiSi were also statistically similar (*P*>.999) (3.99 ±0.81 μm and 3.91 ±0.85 μm). The roughness of the additively manufactured ZrO_2 was statistically similar after airborne-particle abrasion (*P*=.549) (0.39 ±0.50 μm and 0.79 ±0.98 μm). The roughness of the treated subtractively manufactured ZrO_2 was in a comparable range at 0.82 ±0.76 μm having been 0.67 ±0.73 μm previously, (*P*=.997).

Table 1. Mean ±standard deviation TBS values for different testing groups

Groups	TBS Additive [MPa]	TBS Subtractive [MPa]
ZrO_2-Dentin	5.88 ±2.22	6.30 ±2.74
ZrO_2- ZrO_2	12.40 ±1.56	14.23 ±3.22
LiSi-Dentin	6.34 ±2.26	6.26 ±2.42
LiSi-LiSi	16.27 ±6.13	18.82 ±5.92

TBS, tensile bond strength.

Table 2. Weibull analysis for different tested groups

Group	*m*	σ_θ in [MPa]	R^2
LiSi-LiSi additive	2.93	18.27	0.976
LiSi-LiSi subtractive	2.90	21.38	0.936
LiSi-Dentin additive	2.97	7.15	0.946
LiSi-Dentin subtractive	2.57	7.11	0.948
ZrO_2- ZrO_2 additive	9.61	13.05	0.940
ZrO_2- ZrO_2 subtractive	5.07	15.49	0.985
ZrO_2-Dentin additive	2.85	6.62	0.977
ZrO_2-Dentin subtractive	2.41	7.16	0.918

M, Weibull module; σ_θ:, characteristic tensile strength

DISCUSSION

The biomechanical and esthetic integrity of ceramic restorations requires a clinically reliable bond to dentin. The μTBS has been reported to be the best surrogate measure of the interfacial bonding effectiveness of dental restorative materials, particularly after subjecting those specimens to durability testing.[20] Nevertheless, limitations of the μTBS have been reported, with a lack of general agreement on test standards, inconsistency between results, difficulties in preparing specimens, and the risk of pretest failures.[20–23] In the current study, the TBS testing protocol avoided pretest failures and confirmed the accurate reproducibility of specimen size with simplified test standards and assembly. Additively manufactured ceramics have not yet been thoroughly investigated. Bonding to dental substrates is subject to variation among teeth but simulates clinical behavior. The bond of ceramic to the corresponding ceramic provides an opportunity to assess the internal material properties, as the adhesive bond was examined without the indeterminable variable tooth in order to have a baseline of the adhesive properties of additively manufactured ceramics.

Both null hypotheses, that the ceramic-ceramic and ceramic-dentin bond strength and surface topography of ZrO_2 and LiSi would not differ between additively and subtractively manufactured ceramics, were not rejected, as significant differences were not found between the manufacturing methods (*P*>.05). When the ceramics were bonded to corresponding ceramics, higher values were achieved than with ceramics to dentin. The highest bond strength values were measured by both additive and subtractive LiSi groups. These results were consistent with those of an investigation that reported

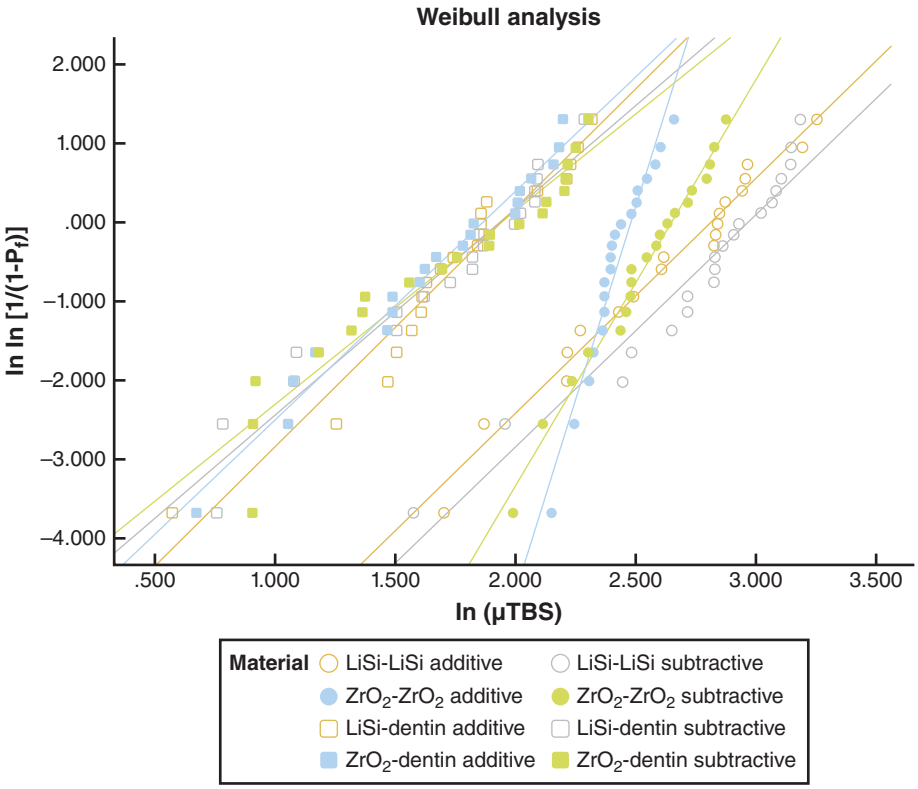

Figure 2. Weibull analysis.

reliable bonding between IPS e.max CAD and the luting composite resin after etching and the use of universal primer.[20] The Weibull moduli for the 2 lithium disilicate groups (2.93 and 2.90) indicated that their adhesive bond had similar reliability. The 3Y-TZP ZrO_2 tested showed lower adhesion values, with Weibull moduli of 9.61 for the additively and 5.07 for the subtractively manufactured ceramics, and achieved the highest

Table 3. Mean ±standard deviation roughness measurement Sa (μm) for ceramic type before and after surface treatment

Material	Untreated Sa in (μm)	Treated (HF/ Airborne-particle Abrasion) Sa in (μm)
LiSi additive	3.70 ±0.60	3.90 ±0.28
LiSi subtractive	3.99 ±0.81	3.91 ±0.85
ZrO_2 additive	0.39 ±0.50	0.79 ±0.98
ZrO_2 subtractive	0.67 ±0.73	0.82 ±0.76

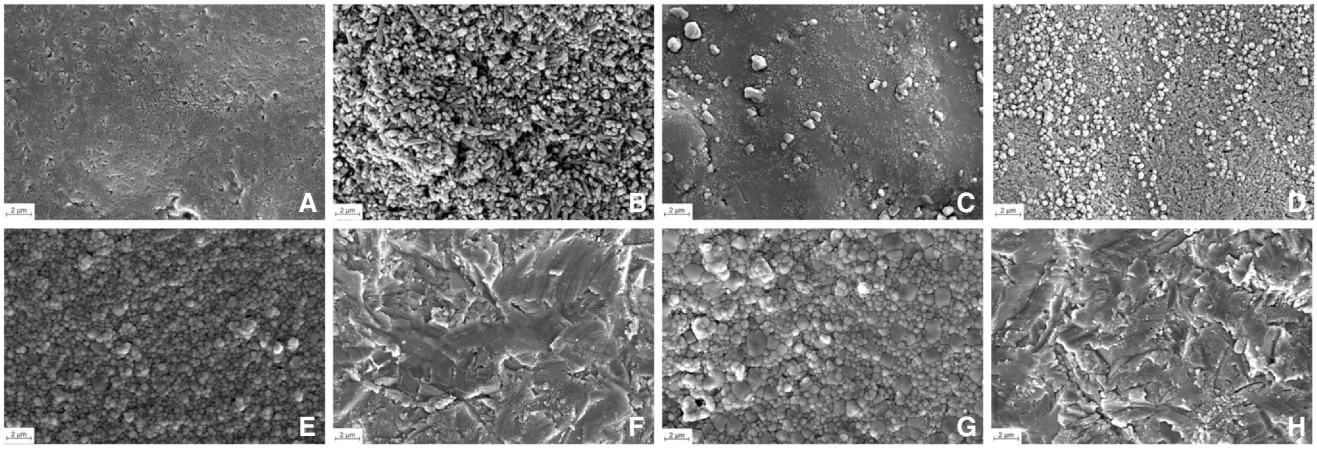

Figure 3. Scanning electron microscope images (original magnification ×4000). A, Lithium disilicate additive untreated. B, Lithium disilicate additive etched. C, Lithium disilicate subtractive untreated. D, Lithium disilicate subtractive etched. E, Zirconia additive untreated. F, Zirconia additive airborne-particle abraded. G, Zirconia subtractive untreated. H, Zirconia subtractive airborne-particle abraded.

measured reliability. The results were consistent with those of an earlier study confirming that different ZrO_2 materials exhibit no significant difference regarding bond strength (P>.05).[40]

All fractures were cohesive within the luting composite resin in the ceramic-ceramic specimens; therefore, the bond strength to ceramic was higher than the inherent strength of the substrates, in particular the luting composite resin.[24] This result is important, as recommendations for the adhesive placement of additively manufactured ZrO_2 and LiSi restorations are lacking. Based on the results, conditioning the additive manufactured ceramic with HF for 30 s in combination with silane for LiSi and airborne-particle abrasion with Al_2O_3 at 0.1 to 0.2 MPa in combination with MDP primer for ZrO_2 can be recommended. When the ceramics were bonded to dentin, the bond strength was lower, as expected, and the fracture pattern was adhesive failure. The weakest point shifted from the luting composite resin to the adhesion between dentin and adhesive.[27–29] No significant differences between the manufacturing methods was found (P>.05). In the Weibull diagram (Fig. 2), the graphs are close to each other, indicating high reliability, regardless of the manufacturing process.[39]

The layered building of additively manufactured objects can lead to anisotropic mechanical properties, as reported previously.[6,7,16] Low strength interfaces or interlayer porosity between the 2D layers can facilitate the fracture path and delamination. Even though the layers were aligned perpendicular to the tensile force, no evidence for anisotropic behavior, such as delamination, was found in the observed fracture patterns. The postprocessing of additive ceramic involves a 2-stage heat treatment after the green parts are cleaned to convert them into ceramic parts with a density above 99.9%.[14,34]

No significant differences were observed using digital microscope measurement (P>.05) with or without LiSi etching in the subtractive or additive group. In this regard, the literature is highly diverse, and evidence indicates that the surface is enhanced[30] and no significant difference occurs after etching (P>.05).[31,32] However, a higher surface roughness has been reported not to be directly associated with a higher adhesive strength.[33] In addition, the roughness depends on the manufacturing process, especially for subtractive processes. Differences in CAD-CAM milling processes, such as milling tools, milling process settings, and milling environment (wet or dry), lead to different results of the measured surface roughness. The mean values of ZrO_2 confirm that airborne-particle abrasion with Al_2O_3 increases the surface roughness of ZrO_2 ceramics.[36,37]

Scanning electron microscope images can provide further insight into the difference between the materials. The crystals of the additively manufactured LiSi group were observed to be angular, pointed, and rod-shaped. In comparison, the crystals of the subtractively produced group were platelet-shaped and slightly smaller. The reason for the difference in the additive group can be found in the manufacturing process and the LiSi crystals used in the initial state when ceramic blocks are ground to powder. Baumgartner et al[13] used a fine powder consisting of ground e.max pressed blanks for the slurry and could achieve comparable with and even higher mechanical properties than those of conventionally produced IPS e.max ceramic. To ensure proper coating behavior during the printing process, the viscosity of the slurry should not exceed 20 Pas; otherwise, failures, such as voids between the printing layers or total misprints, will occur.[12] The crystal structure of the LiSi ceramic is therefore dependent on the raw material, and the crystal size on the sintering temperature.[13]

After the ZrO_2 was airborne-particle abraded with Al_2O_3, the grain boundaries in both the additive and subtractive groups disappeared, and the typical morphology of yttrium-stabilized zirconia was no longer visible. The kinetic energy of the alumina particles during airborne-particle abrasion has been reported to be high enough to cause the surface of zirconia-based materials to melt.[41] In addition, defects in the form of microcracks and plastic deformations can be seen in the images. These defects usually occur because of internal stresses and the increased temperatures caused by the impact of Al_2O_3 particles. However, the surface changes appear in a comparable form in both additively and subtractive manufactured ZrO_2, and these defects have been described as a common phenomenon in any type of ZrO_2.[41,42] Furthermore, the defects caused by airborne-particle abrasion could be repaired by filling the defects and "healing" the surface with luting composite resins.[43,44]

As often discussed, the μTBS testing has limitations but remains a valid method of assessing bonding effectiveness, especially by evaluating ceramics.[25,26] The small specimen size (4 mm^2) provided minimum scatter in the results; however, the bonding process is technique sensitive. Aging in the oral environment can be simulated by thermocycling. Due to a lack of standardization, it is difficult to compare our results with those of other studies.

Digital additive manufacturing workflows allow the manufacture of multiple restorations in parallel with different color and material gradients and with highly complex geometries. However, the acceleration and simplification of the postprocessing workflow will be an important goal in facilitating its introduction into dentistry.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Additive manufacturing of the tested ceramics did not cause a significant difference in the bond strength compared with subtractive manufacturing.

2. The weakest point in bonding was the interlayer between the composite resin and dentin.
3. The recently introduced printed materials showed similar reliability to the conventionally milled materials.

REFERENCES

1. Spitznagel FA, Boldt J, Gierthmuehlen PC. CAD/CAM ceramic restorative materials for natural teeth. *J Dent Res*. 2018;97:1082–1091.
2. Kessler A, Hickel R, Reymus M. 3D printing in dentistry-state of the art. *Oper Dent*. 2020;45:30–40.
3. Wang H, Aboushelib MN, Feilzer AJ. Strength influencing variables on CAD/CAM zirconia frameworks. *Dent Mater*. 2008;24:633–638.
4. Jadhav A, Jadhav VS. A review on 3D printing: An additive manufacturing technology. *Mater Today Proc*. 2022;62:2094–2099.
5. Paolini A, Kollmannsberger S, Rank E. Additive manufacturing in construction: A review on processes, applications, and digital planning methods. *Addit Manuf*. 2019;30:100894.
6. Es-Said OS, Foyos J, Noorani R, et al. Effect of layer orientation on mechanical properties of rapid prototyped samples. *Mater Manuf Process*. 2000;15:107–122.
7. Keßler A, Hickel R, Ilie N. In vitro investigation of the influence of printing direction on the flexural strength, flexural modulus and fractographic analysis of 3D-printed temporary materials. *Dent Mater J* 2021.
8. Brinckmann SA, Young JC, Fertig RS, Frick CP. Effect of print direction on mechanical properties of 3D printed polymer-derived ceramics and their precursors. *Mater Lett: X*. 2023;17:100179.
9. Marcus HL, Beaman JJ, Barlow JW, Bourell DL. Solid freeform fabrication-powder processing. *Am Ceram Soc bull*. 1990;69:1030–1031.
10. Sachs E, Cima M, Cornie J. Three-dimensional printing: Rapid tooling and prototypes directly from a CAD model. *CIRP Ann Manuf Technol*. 1990;39:201–204.
11. Chen Z, Li Z, Li J, et al. 3D printing of ceramics: A review. *J Eur Ceram Soc*. 2019;39:661–687.
12. Gmeiner R. Stereolithographic ceramic manufacturing (SLCM) of lithium disilicate and bioactive glass ceramics for dental and medical applications [dissertation]. *Technische Universität Wien* 2016.
13. Baumgartner S, Gmeiner R, Schönherr JA, Stampfl J. Stereolithography-based additive manufacturing of lithium disilicate glass ceramic for dental applications. *Mater Sci Eng C Mater Biol Appl*. 2020;116:111180.
14. Schwentenwein M, Schneider P, Homa J. Lithography-based ceramic manufacturing: A novel technique for additive manufacturing of high-performance ceramics. *Adv Sci Technol*. 2014;88:60–64.
15. Dehurtevent M, Robberecht L, Hornez JC, et al. Stereolithography: A new method for processing dental ceramics by additive computer-aided manufacturing. *Dent Mater*. 2017;33:477–485.
16. Nakai H, Inokoshi M, Nozaki K, et al. Additively manufactured zirconia for dental applications. *Materials (Basel)*. 2021;14:3694.
17. Dönmez MB, Yucel MT, Kilic I, Okutan Y. Novel ceramic primer vs. conventional treatment methods: Effects on roughness and bond strength of all-ceramic restorations. *Am J Dent*. 2018;31:249–252.
18. Souza JCM, Raffaele-Esposito A, Carvalho O, et al. Surface modification of zirconia or lithium disilicate-reinforced glass ceramic by laser texturing to increase the adhesion of prosthetic surfaces to resin cements: An integrative review. *Clin Oral Investig*. 2023;27:3331–3345.
19. Gorman CM, de Faoite D, Flannery D, et al. Alteration of the intaglio surface of lithium disilicate glass-ceramic. *J Prosthet Dent*. 2019;122:411.e1–411.e10.
20. Van Meerbeek B, Peumans M, Poitevin A, et al. Relationship between bond-strength tests and clinical outcomes. *Dent Mater*. 2010;26:e100–e121.
21. Armstrong S, Geraldeli S, Maia R, et al. Adhesion to tooth structure: A critical review of "micro" bond strength test methods. *Dent Mater*. 2010;26:e50–e62.
22. De Munck J, Van Landuyt K, Peumans M, et al. A critical review of the durability of adhesion to tooth tissue: Methods and results. *J Dent Res*. 2005;84:118–132.
23. Sano H, Chowdhury A, Saikaew P, et al. The microtensile bond strength test: Its historical background and application to bond testing. *Jpn Dent Sci Rev*. 2020;56:24–31.
24. Liebermann A, Detzer J, Stawarczyk B. Impact of recently developed universal adhesives on tensile bond strength to computer-aided design/manufacturing ceramics. *Oper Dent*. 2019;44:386–395.
25. Della Bona A, van Noort R. Shear vs. tensile bond strength of resin composite bonded to ceramic. *J Dent Res*. 1995;74:1591–1596.
26. Inokoshi M, De Munck J, Minakuchi S, Van Meerbeek B. Meta-analysis of bonding effectiveness to zirconia ceramics. *J Dent Res*. 2014;93:329–334.
27. Aker Sagen M, Vos L, Dahl JE, Ronold HJ. Shear bond strength of resin bonded zirconia and lithium disilicate - Effect of surface treatment of ceramics and dentin. *Biomater Investig Dent*. 2022;9:10–19.
28. Sagen MA, Kvam K, Ruyter EI, Ronold HJ. Debonding mechanism of zirconia and lithium disilicate resin cemented to dentin. *Acta Biomater Odontol Scand*. 2019;5:22–29.
29. Peumans M, Valjakova EB, De Munck J, et al. Bonding effectiveness of luting composites to different CAD/CAM materials. *J Adhes Dent*. 2016;18:289–302.
30. Zogheib LV, Bona AD, Kimpara ET, McCabe JF. Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. *Braz Dent J*. 2011;22:45–50.
31. May MM, Fraga S, May LG. Effect of milling, fitting adjustments, and hydrofluoric acid etching on the strength and roughness of CAD-CAM glass-ceramics: A systematic review and meta-analysis. *J Prosthet Dent*. 2022;128:1190–1200.
32. Prochnow C, Venturini AB, Grasel R, et al. Effect of etching with distinct hydrofluoric acid concentrations on the flexural strength of a lithium disilicate-based glass ceramic. *J Biomed Mater Res B Appl Biomater*. 2017;105:885–891.
33. Xiaoping L, Dongfeng R, Silikas N. Effect of etching time and resin bond on the flexural strength of IPS e.max Press glass ceramic. *Dent Mater*. 2014;30:e330–e336.
34. Schönherr JA, Baumgartner S, Hartmann M, Stampfl J. Stereolithographic additive manufacturing of high precision glass ceramic parts. *Materials (Basel)*. 2020;13:1492.
35. Armstrong S, Breschi L, Ozcan M, et al. Academy of dental materials guidance on in vitro testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (muTBS) approach. *Dent Mater*. 2017;33:133–143.
36. Yang B, Barloi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. *Dent Mater*. 2010;26:44–50.
37. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dent Mater*. 2007;23:45–50.
38. El Gezawi M, Haridy R, Abo Elazm E, et al. Microtensile bond strength, 4-point bending and nanoleakage of resin-dentin interfaces: Effects of two matrix metalloproteinase inhibitors. *J Mech Behav Biomed Mater*. 2018;78:206–213.
39. Quinn JB, Quinn GD. A practical and systematic review of Weibull statistics for reporting strengths of dental materials. *Dent Mater*. 2010;26:135–147.
40. Le M, Larsson C, Papia E. Bond strength between MDP-based cement and translucent zirconia. *Dent Mater J*. 2019;38:480–489.
41. Okada M, Taketa H, Torii Y, et al. Optimal sandblasting conditions for conventional-type yttria-stabilized tetragonal zirconia polycrystals. *Dent Mater*. 2019;35:169–175.
42. Kim HK, Ahn B. Effect of Al(2)O(3) sandblasting particle size on the surface topography and residual compressive stresses of three different dental zirconia grades. *Materials (Basel)*. 2021;14:610.
43. Burke FJ, Fleming GJ, Nathanson D, Marquis PM. Are adhesive technologies needed to support ceramics? An assessment of the current evidence. *J Adhes Dent*. 2002;4:7–22.
44. Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. *J Prosthet Dent*. 2004;91:356–362.

Corresponding author:
Dr Andreas Kessler
Ludwig-Maximilian-University of Munich
Department of Conservative Dentistry and Periodontology
Goethestrasse 70
Munich 80336
GERMANY
Email: andreas.kessler@med.uni-muenchen.de

CRediT authorship contribution statement

Andreas Kessler: Conceptualization, Methodology, Project administration, Resources, Supervision, Roles/writing - original draft, Writing - review and editing. **Thilo Reichert**: Data curation, Investigation, Roles/writing - original draft. **Stefanie Lindner**: Formal analysis, Methodology, Writing - review and editing. **Anja Liebermann**: Conceptualization, Methodology, Supervision, Writing - review and editing. **Moataz El Gezawi**: Supervision, Writing - review and editing. **Dalia Kaisarly**: Conceptualization, Methodology, Project administration, Supervision, Roles/writing - original draft, Writing - review and editing.

https://doi.org/10.1016/j.prosdent.2024.04.002