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Flexural Strength of two Different Ceramic Materials Bonded to CAD/CAM Titanium (In Vitro Study)

Yousreya Shalaby PhD[1], Dawlat Mostafa PhD[2,3] and Lamiaa Hamdy BDS[4]

[1,4]Department of Conservative Dentistry (Fixed Prosthesis), Faculty of Dentistry, Alexandria University, Egypt.
[2]Department of Dental Biomaterials, Faculty of Dentistry, Alexandria University, Egypt.
[3]College of Dentistry, The Arab Academy for Science and Technology and Maritime Transport (AASTMT)/El-Alamein, Egypt.
yashalaby@yahoo.com; dwlat.mostafa@yahoo.com; lamiaahamdy1204@gmail.com

Abstract: Introduction: Metal-Ceramic bonding could be considered as a crucial issue for efficacious dental restorations. Zirconia is a promising ceramic alternative to porcelain in modern dentistry. **Objectives:** The purpose of this study was to investigate the flexural strength of low fusing porcelain and CAD/CAM zirconia materials bonded to CAD/CAM titanium by the effect of sandblasting with 50µm aluminum oxide and application of bonding agent. **Materials and Methods:** A CAD/CAM machine was used to mill 36 titanium bar specimens of Grade 2 titanium in the dimensions of (25.0 mm × 3.0 mm × 0.5 mm). The specimens were divided into two equal groups (N=18) and sandblasting was performed to one of these groups using 50µm aluminum oxide. Each of these groups (Sandblasted and non-sandblasted) were further sub grouped into two equal groups (N=9) according to veneering material either low-fusing porcelain or CAD/CAM zirconia. A universal testing machine was used to perform the 3-point bending test. The titanium-porcelain and titanium-zirconia interfaces were subjected to stereomicroscopic as well as scanning electron microscopic analysis. The bond failure data (MPa) were analyzed using Student t-test. **Results:** The debonding test showed that sandblasted subgroups veneered with either low fusing porcelain or zirconia resulted in the strongest titanium-ceramic bond (29.09 ± 2.69 MPa and 32.41 ± 1.29 MPa). Whereas, non-sandblasted subgroups veneered with both low fusing porcelain and zirconia resulted in unsatisfactory bond strength (13.14± 1.93 and 12.27 ± 1.90). The photomicrographs of the titanium surface after debonding demonstrated more residual porcelain retained on the metal surface for sandblasted subgroups. **Conclusions:** Sandblasting with alumina produced a significant increase in the bond strength between titanium and the veneering material while application of bonding agent alone without sandblasting resulted in insufficient bond strength. Bonding between zirconia and titanium by means of bonding agent in the presence of sandblasting showed significant results comparable to that of low fusing porcelain.

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1. Introduction

Titanium and its alloys have a number of desirable properties that would recommend them for dental use, including excellent biocompatibility, corrosion resistance, low specific gravity, mechanical properties that are comparable to those of dental gold alloys, and relatively low cost (1). Titanium has a variety of applications in dentistry including endosseous implants as well as removable and fixed partial dentures (2-4). Nevertheless, many practical problems with the use of titanium and titanium alloys for dental crown and fixed partial denture applications remain unsolved. Titanium reacts strongly with gaseous elements such as oxygen at high temperature, and yields an excessively thick layer of TiO_2. Such an oxide layer is considered a detriment to titanium-porcelain bonding, as it can easily break or spall. The metal oxide layer should be a monolayer in thickness to create an effective metal-ceramic bond. Therefore, it is essential that porcelain firing should occur below 800°C to prevent excessive oxide formation (5-7). Another obstacle in the effective bonding between titanium and porcelain is the great mismatch in the coefficient of thermal expansion (CTE), because CP Ti has a much smaller CTE than do conventional porcelains (8). Accordingly, low-fusing dental porcelains are used for titanium veneering. Previously, titanium was only available for dental use through casting which differs considerably from other dental alloys casting. This is owing to the relatively high melting point of pure titanium, high affinity to other elements such as oxygen, as well as its light weight (9,10). It has also been found that cast titanium restorations are more susceptible to corrosion than machined titanium restorations (11). Currently, copings can be milled using machines from an industrial prefabricated material block, or manufactured with electron discharge in combination with a milling technique (12). This is could be accomplished with modified computer numeric control (CNC) equipment (13) or milled from prefabricated titanium blocks using computer aided design/computer assisted manufacturing (CAD/CAM) systems (14). Copings produced in this manner are characterized by the fine and homogenous structure of titanium without a reactive layer on the surface and require much less time (13). Successful bonding between titanium and porcelain remains an area of research and numerous surface treatments were proposed throughout the literature to enhance their bonding strength. This can be proficient by increasing wettability of the metal surface, which can be made by increasing surface energy (15). Although many physical and chemical surface treatments of Ti have been suggested such as: sandblasting with alumina combined with steam cleaning or ultrasonic cleaning (16), coating with Au (17), silicon nitride (18), Cr and ceramic (19), acid treatments (HF, H_2SO4, and H_2O_2), or other surface modification techniques (Silicoater, Silicoater MD, Rocatec, Siloc Systems), Mechanical bonding remains a pillar in a good clinical performance of metal-ceramic prostheses. Although porcelain has been used traditionally for veneering of titanium copings, zirconia could be proposed as a promising alternative to porcelain. This is attributed to the excellent properties of zirconia compared to porcelain, including good chemical and dimensional stability, mechanical strength and toughness, coupled with a Young's modulus in the same order of magnitude as stainless steel alloys (20). The three-point flexure test could be considered a convenient technique for bond strength measuring of ceramo-metal samples as it provides complex tensions on the samples during the test (21). From this viewpoint, the current in-vitro study was an attempt to evaluate the flexural strength of low fusing porcelain and CAD/CAM zirconia materials bonded to CAD/CAM titanium by the effect of sandblasting with 50μm aluminum oxide and application of bonding agent.

2. Materials and Methods

2.1. Materials

The materials used in the present study were CAD/CAM titanium blocks (EVEREST T-Ronde Grade 2), Low fusing porcelain for titanium (Noritake Super Porcelain Ti-22; Noritake Dental Supply Co Ltd, Nagoya, Japan), Bonding porcelain liquid (Ti-22 BONDING PORCELAIN: Zirconzahn, Italy) and CAD/CAM zirconia blocks (prettau zirconia).

2.2. Methods

2.2.1. Grounding of Titanium (Ti) Bars

Thirty-six titanium bars (N=36) in the dimensions of (25 mm × 3 mm × 0.5 mm) were milled from CAD/CAM titanium blocks (EVEREST T-Ronde Grade 2) using CAD/CAM machine (KaVo Everest, Germany) according to the international standard 9693-1 for flexural-strength test metal substructure specimens (22). Milled Ti bars were separated from the CAD/CAM blank, then each specimen was finished and polished using different sizes of carbide burs and discs on low speed hand piece. All prepared bars were then checked for correct dimensions using an endodontic ruler for length and width and a gauge for thickness.

2.2.2. Sandblasting of Titanium (Ti) Bars

Milled titanium bars (N=36) were randomly divided into two equal groups (N=18) and sandblasting was performed to one of these groups by mounting each Ti bar on a special holder with the nozzle of the sandblasting device fixed at 2 mm from the center of the specimen at a 90^o incidence angle. Sandblasting was performed using 50μm aluminium oxide at 0.5Mpa air pressure for 10 s. Following sandblasting, All the specimens (N=36) were then ultrasonically cleaned using acetone solution for 10 minutes and finally received an oxidation treatment through Firing at 500°C to 800°C with the heat rate of 50°C/min followed by a holding time of 3min. under vacuum of 99kPa（74cm/Hg).

2.2.3. Grouping

The thirty-six milled titanium bars (N=36) were equally separated according to the sandblasting surface treatment into two groups (N=18); Sandblasted Ti bars and Non-sandblasted group. Subsequently, each of the previous two groups was subdivided into two equal subgroups according to the used veneering material resulting in four subgroups (N=9) as follows; Sandblasted Ti bars veneered with low fusing porcelain (TSP), Non-sandblasted Ti bars veneered with low fusing porcelain (TNSP), sandblasted Ti bars veneered with CAD/CAM zirconia (TSZ) and Non-sandblasted Ti bars veneered with CAD/CAM zirconia (TNSZ).

2.2.4. Titanium Veneering Protocol

2.2.4.1. Titanium veneering with low-fusing porcelain

Low fusing porcelain (*Noritake Super Porcelain; Noritake Dental Supply Co Ltd, Nagoya, Japan*) was applied according to manufacturer’s instructions using layering technique; Bonding porcelain (BP), opaque porcelain and body porcelain. BP was mixed with BP liquid and the mixture was applied using a brush on the porcelain bonding surface in the central portion of each metal strip with a length of 8mm and width of 3mm yielding a final thickness of 0.2mm thickness.

Following BP application, Opaque porcelain was applied in approx. 0.15mm thickness (in the middle on the previously applied BP) to obtain desired shade. Finally, body porcelain was applied with the same dimensions as opaque porcelain. The firing shrinkage was compensated for by applying a second layer of body porcelain, yielding a final total thickness of 1mm.

2.2.4.2. Titanium veneering with CAD/CAM zirconia

Eighteen zirconia bar specimens were milled from CAD/CAM zirconia blocks (prettau zirconia, Zirconzahn, Italy) using Roland DWX50 milling machine. Specimens were separated from the blank using a bur mounted on straight hand piece, and smooth surfaces were attained, followed by sintering according to manufacturer's instructions. Shrinkage was calculated so that the final dimensions of each specimen was 8mm length ×3mm wide× 1mm thickness. Afterward, milled zirconia specimens were bonded to veneer milled Tibars by means of bonder. BP was mixed with BP liquid and the mixture was applied on the central portion of each metal strip using a brush with a length of 8mm and width of 3mm. Milled zirconia specimens were fixed on the applied bonder and baked according to manufacturer's instructions.

2.2.5. Flexural Strength Test

The flexural strength test was performed to the previously mentioned four tested subgroups according to international standard 9693-1 using a three-point bending test on a universal testing machine (AGS-X 5 KN, shimadzu, Japan) equipped with a 5-kN load cell and crosshead speed of 0.5 mm/min. The specimens were positioned on a specially fabricated metal support to align and stabilize the specimens with porcelain and zirconia facing downward. The load was applied at the midpoint of the metal strip with a rounded-tip loading knife until a sudden drop in load occurred in the load-deflection curve, indicating the bond failure. The failure load was recorded digitally using software provided by the manufacturer of the testing machine. The bond strength (s) was calculated by the following equation given in ISO 9693:

$$\sigma = k.F \ (N/mm^2)$$

Where F is the maximum force applied in New tons before debonding (failure load), and k is a constant determined from a graph in ISO 9693 with units of mm^{-2}. The value of k depends on the thickness of the metal substrate and the elastic modulus of the metallic material, and for the commercially pure titanium tested it was determined to be 4.6 mm^{-2}.

2.2.6. Surface Morphology and Bond Failure Assessment

Following flexural strength testing, surfaces of titanium where debonding of porcelain and zirconia occurred (failure interface) were examined and captured by stereomicroscope (Olympus, Japan). Representative specimens of each subgroup were chosen to be further analyzed using scanning electron microscope (JEOL JSM 636OLA Analytical Scanning Electron, USA) at a magnification of X15 and 20.0 kv to identify the failure pattern and determine the micro morphological topography. Selected specimens were gold coated, and introduced to the vacuum chamber. Areas of interest were captured and recorded.

2.2.7. Statistical Analysis

The Data was collected and entered into the personal computer. Statistical analysis was done using Statistical Package for Social Sciences (SPSS/version 20) software. Arithmetic mean, standard deviation, for two groups t-test was used. The level of significant was 0.05.

3. Results

3.1. Flexural Strength

Regarding the flexure strength results, means and SD of flexure bond strength in (Mpa) are showed in tables (1-4). There was a providentially higher statistically significant difference between flexural strength values of sandblasted surface treated specimens and non-sandblasted subgroups for both low fusing porcelain and zirconia veneers ($p < 0.001$). Conversely, no statistically significant differences were detected between low fusing porcelain and zirconia veneers either for subgroups with sandblasting surface treatments ($p = 0.006$) or non-sandblasted subgroups ($p = 0.350$).

Table (1): Comparison between flexural strength values (MPa) of low fusing porcelain sandblasted treated surfaces and non-sandblasted surfaces.

Flexural strength	**Low fusing porcelain**		**t**	**p**
	(TSP) (n = 9)	**(TNSP) (n = 9)**		
Min. – Max.	26.02 – 34.40	10.32 – 16.44		
Mean ± SD.	29.09 ± 2.69	13.14± 1.93	14.467^*	$<0.001^*$
Median	28.40	13.27		

t: Student t-test p: p value for comparing between the studied groups *: Statistically significant at $p \leq 0.05$

Table (2): Comparison between zirconia sandblasted and non-sandblasted surfaces according to flexural strength in MPa.

Flexural strength	Zirconia		t	p
	(TSZ) (n = 9)	(TNSZ) (n = 9)		
Min. – Max. Mean ± SD. Median	30.30 – 34.51 32.41 ± 1.29 32.45	9.58 – 14.68 12.27 ± 1.90 12.90	26.305^*	$<0.001^*$

t: Student t-test p: p value for comparing between the studied groups *: Statistically significant at $p \leq 0.05$

Table (3): Comparison between flexural strength values (MPa) of sandblasted low fusing porcelain and zirconia veneers.

Flexural strength	Sandblasted Treated Veneers		t	p
	(TSP) (n = 9)	(TSZ) (n = 9)		
Min. – Max. Mean ± SD. Median	26.02 – 34.40 29.09 ± 2.69 28.40	30.30 – 34.51 32.41 ± 1.29 32.45	3.338^*	0.006^*

t: Student t-test p: p value for comparing between the studied groups *: Statistically significant at $p \leq 0.05$

Table (3): Comparison between flexural strength values (MPa) of non-sandblasted low fusing porcelain and zirconia veneers.

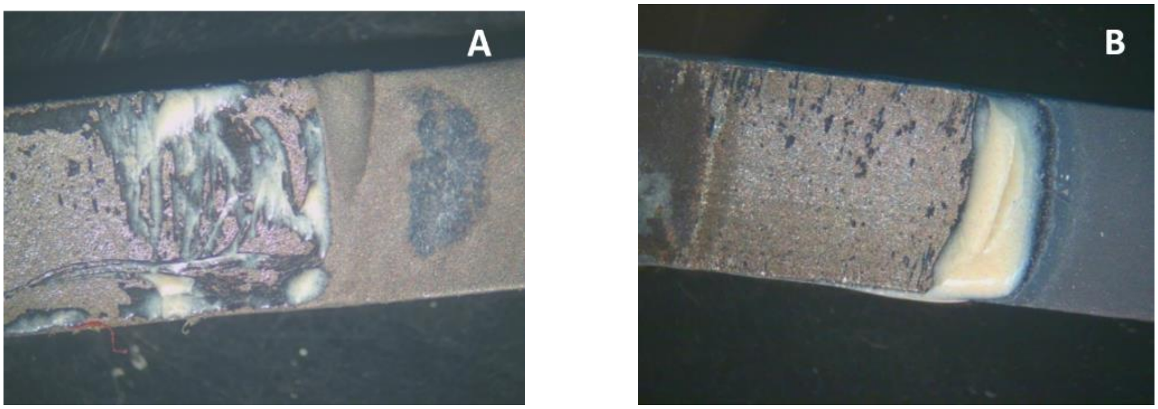
Flexural strength	Non-sandblasted Treated Veneers		t	p
	(TSP) (n = 9)	(TSZ) (n = 9)		
Min. – Max. Mean ± SD. Median	10.32 – 16.44 13.14± 1.93 13.27	9.58 – 14.68 12.27 ± 1.90 12.90	0.963	0.350

t: Student t-test p: p value for comparing between the studied groups *: Statistically significant at $p \leq 0.05$

3.2. Surface Morphology and Bond Failure Imaging

3.2.1. Bond Failure with Stereomicroscope

Stereomicroscope examination revealed that air born particle abrasion with 50-µm aluminum oxide in both TSP and TSZ subgroups increased the surface roughness of the titanium surface resulting in increased areas of retained ceramic (Fig.1: A & B). On the other hand, TNSP and TNSZ surfaces appeared to be smooth with limited areas of retained ceramic (Fig.1: C & D).



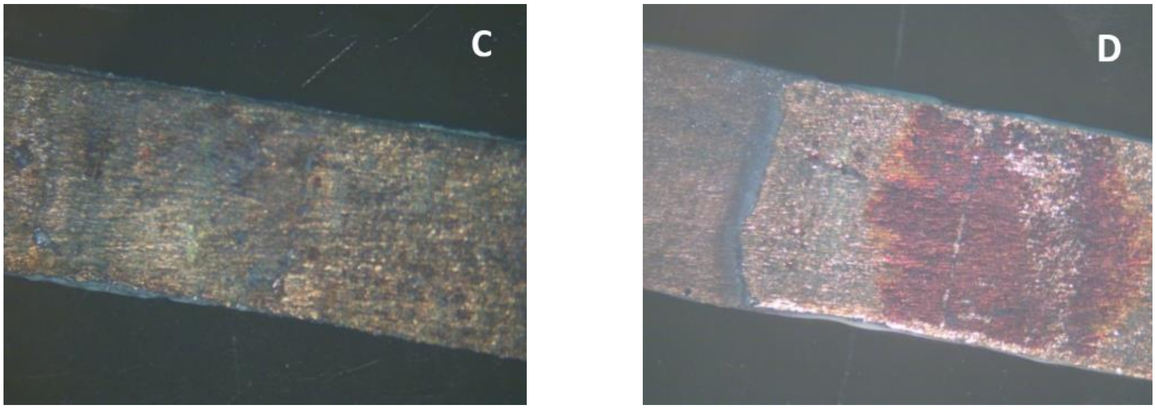


Figure 1: (A-D): Stereomicroscopic images for low fusing porcelain and zirconia veneers subgroups. Sandblasted treated surfaces showed areas of retained bonder mainly in the central regions of TSP surfaces (image A) and in the periphery of TSZ surfaces (image B). While, smooth surfaces without any retained bonder were displayed in the TNSP (image C) and TNSZ (image D) subgroups.

3.2.2. Surface Morphology and Bond Failure with SEM

The photomicrographs of the milled titanium surfaces after debonding demonstrated residual porcelain retained on the metal surface for all subgroups. This observation indicates a combination of cohesive and adhesive bond failures. However, more traces of porcelain were observed on sandblasted specimens treated with airborne-particle abrasion (TSP and TSZ subgroups), which may indicate that surface treatment with alumina has resulted in increasing surface roughness of titanium and thus increased bonding (Fig.2: A & B). Untreated subgroups (TNSP and TNSZ) revealed very few residual porcelains remaining (Fig. 2: C & D). This illustrates that failure was primarily adhesive for the untreated subgroups.

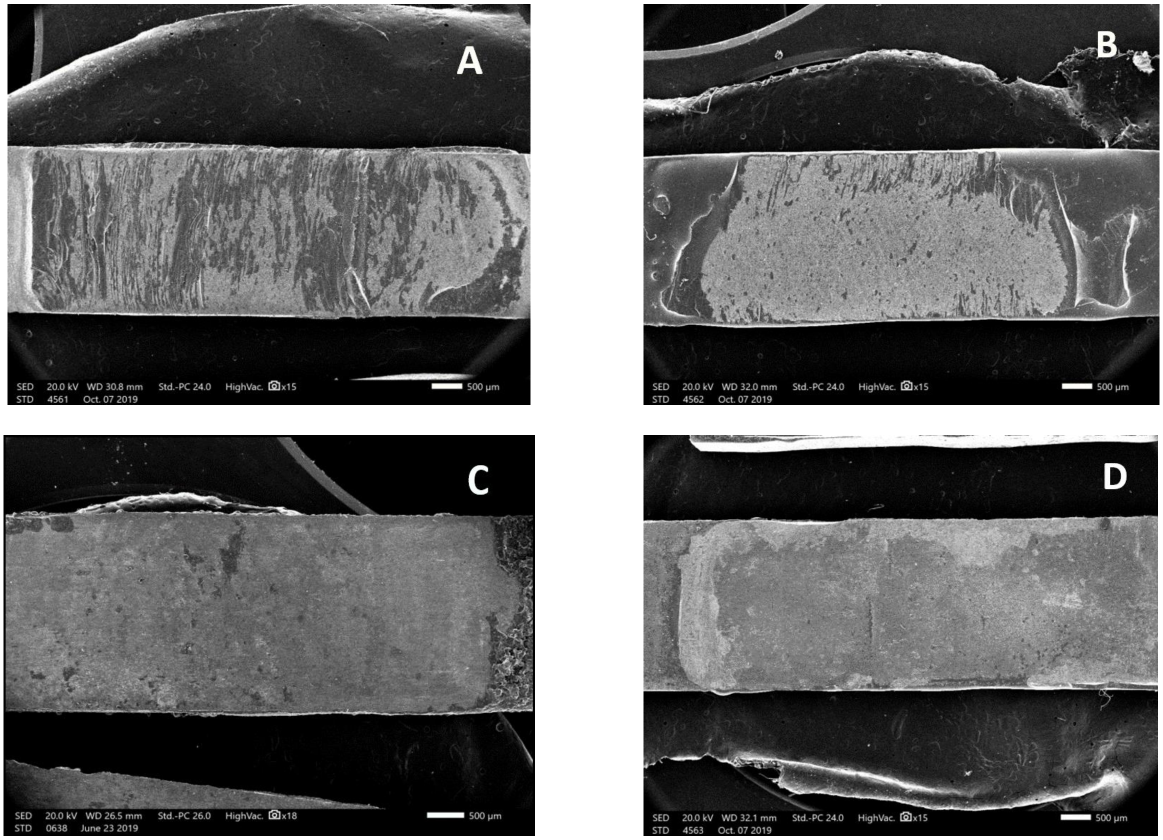


Figure 2: (A-D): SEM images showing the failed surfaces of low fusing porcelain and zirconia veneers. Dark areas represent retained ceramic and light areas represent titanium. (Original magnification X15). A: TSP surfaces, B: TSZ surfaces, C: TNSP surfaces and D: TNSZ surfaces.

4. Discussion

The use of titanium in dentistry in recent years has increased dramatically because of its superior biocompatibility, corrosion resistance, desirable physical and mechanical properties, and relatively low cost compared to other metals (13, 23-25). Titanium has high affinity to gaseous elements such as oxygen at high temperatures resulting in a series of reactions that produces a thick layer of TiO_2(5,6). Therefore, the thickness of this metal oxide layer should be controlled to create an effective metal-ceramic bond (7). For this reason, low-fusing dental porcelains (with a firing temperature of 800°C) are used for veneering of titanium. Another possible source of conventional porcelain titanium bond failure is the stress caused by the mismatch of the thermal expansion coefficient of titanium and ceramic that may affect the flexural bond strength of the titanium ceramic system. Dental ceramics with coefficient of thermal expansion values less than 8.5x $10^{-6\circ}$C are appropriate for titanium-ceramic restorations (26). Accordingly, low-fusing porcelain was selected in this study as a traditional veneering material to be compared with the high mechanically performing zirconia veneer. Sandblasting with alumina particles is the most common method recommended for creating surface roughness and providing mechanical interlocking force for porcelain. A variety of particle sizes is available for sandblasting of titanium including 50 µm, 100 µm or 250 µm. Previous studies demonstrated that a substantial amount of alumina is always retained on the sandblasted surfaces of alloys. Different studies have reported significantly higher metal-ceramic bond strengths for surfaces sandblasted with 250 µm-grit alumina particles (27,28). However, other studies have documented that fragment retention decreased with increasing grit size (29,30). Al Jabbari *et al*, reported that Alumina particles retained in cp-Ti after sandblasting was found to be independent of grit size due to high ductility of Ti and so 50 µm grit size and 0.5 MPa pressure were recommended for titanium veneered with Ti-22 dental porcelain (32). According to manufacturer's recommendations and Al Jabbari, sandblasting with size 50 µm alumina was selected as a surface treatment method in this current study. From this prospective, titanium bars were veneered by two different ceramic materials; low-fusing porcelain and zirconia, then each veneering material was applied once to sandblasted titanium specimens (using 50 µm alumina particles) and once to non-sandblasted specimens to serve during this in vitro study. Compared to other tests such as shear, torsion and pull through tests, the three-point-flexure test has been advantageous for providing complex tensions on the samples during the tests, being indicated for bond strength measuring of ceramo-metal samples (21). Therefore, it was decided in this existing study to perform three-point flexure test for bond failure assessment according to Probster et al (33). Bonding agent was also applied to titanium prior to veneering in both groups because it is believed that bonding agent plays an important role in titanium-ceramic bond strength by preventing the formation of a nonadherent oxide layer, which is otherwise formed when titaniumis exposed to high temperatures (34, 35). Bonding porcelain (Noritake Ti22) has been reported to have a positive effect on the mean value of the metal-porcelain bond strength (36, 37). It was also reported to have significantly higher bond strength than the other systems as reported by Atsu S. and A car A (36,38). Thus, this in-vitro study was conducted to evaluate the flexural strength of two different veneering materials (low fusing porcelain and CAD/CAM zirconia) bonded to CAD/CAM titanium upon the effect of sandblasting with 50µm aluminum oxide and application of bonding agent. The titanium-ceramic bond of TSP subgroup was 29.09 ± 2.69 compared to that of TNSP subgroup was 13.14± 1.93, whereas for TSZ subgroup the flexure strength mean value in MPa was32.41 ± 1.29 and12.27 ± 1.90 for TNSZ subgroup. It was documented that, the flexural strength results of TNSP subgroup, which had no surface treatment but received a bonding agent were far below the ISO requirement hence it could be indicated that non-sandblasted titanium surfaces may lead to an unsatisfactory titanium-ceramic bond. This result is consistent with the results of previous studies reporting that sandblasting of titanium improves the adhesion of porcelain (35,38,39). Reyes *et al*. reported that Airborne-particle abrasion potentially improves the bond strength by removing loosely attached furrows, overlaps, and flakes of metal created by grinding procedures, provides mechanical interlocking, increases surface area, and increases wettability (35). Regarding to TSP subgroup, surface roughness combined with bonding agent application produced a significant increase in the mean and SD of flexure strength values (29.09 ± 2.69 Mpa) indicating that sandblasting with alumina is a crucial factor in bond strength improvement. Al Hussaini *et al*. stated that using of surface airborne-particle abrasion along with an appropriate bonding agent provided the highest bond strength of the porcelain to the titanium tested, which is in agreement with the present study (37). Concerning zirconia subgroups, the flexural strength results were comparable to that of porcelain subgroups. A significant difference was found when comparing flexure strength of low fusing porcelain and zirconia both with sandblasting surface treatment. Consequently, it could be signposted that bonding of zirconia to titanium by means of bonder was successfully achieved. Whereas bonding of zirconia veneer to non-sandblasted untreated titanium specimens resulted in flexural strength values that were far below the ISO requirement. The bond between Noritake bonding agent and zirconiain this study could be explained by the same mechanism of bond between zirconia frameworks and veneering porcelain. Porcelain used for veneering of zirconia frameworks has a slight mismatch in CTE with that of zirconia, with the porcelain's CTEs lightly lower (40). This will produce a desirable residual compressive stress in the veneering ceramic (41). Whereas, when zirconia's CTE is lower than ceramic's, veneer delamination and microcracks may occur (42). In the current study, the coefficient of thermal expansion of bonder is comparable to that of porcelain used for veneering of zirconia cores and slightly lower than that of zirconia. This slight mismatch may be the cause of strong bond between the bonder and zirconia. The exact mechanism of bonding of zirconia to porcelain is still unknown, but based on few studies, the wettability of the ceramic and zirconia surfaces, chemical bonding, and micromechanical interactions play a key role in this regard (43). SEM evaluation is used in this study as it provides information about the surface morphology and provides high-quality images. Photomicrographs of titanium specimens after debonding for both sandblasted subgroups (TSP and TSZ) revealed excessive traces of residual porcelain retained on the metal surfaces indicating a combination of adhesive and cohesive bond failures. Besides, upon scanning of TSZ surfaces, porcelain traces were less compared to TSP surfaces and were confined mainly at the periphery of the bonding area. It could be opportunely stated that, upon comparing the mechanical testing results with the SEM images, the highest flexural strength was recorded for TSZ surfaces followed by TSP subgroup which could be attributed to the fact that bonding porcelain was more adherent to zirconia surface than the metal interface reflecting mixed bond failure mode for both sandblasted surfaces. In contrast, Untreated subgroups (TNSP & TNSZ) revealed very few residual porcelains remaining on the titanium surfaces indicating that bond failure was manly adhesive in association with decreased flexural strength values.

5. Conclusions:

Within the limitations of this study, sandblasting with alumina produced a significant increase in the bond strength between titanium and both veneering material whether low fusing porcelain or zirconia. Application of bonding agent alone without sandblasting resulted in insufficient bond between titanium and both veneering materials. Bonding between zirconia and titanium by means of bonding agent in the presence of sandblasting showed significant mechanical results comparable to that of low fusing porcelain.

Availability of data and materials

All data presented in the manuscript are available for publication.

Competing interests

Yousreya Shalaby, Dawlat Mostafa and Lamiaa Hamdy declare that they have no competing interests.

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Authors' contributions

Author LH prepared the specimens, Author DM conducted the laboratory Characterizations, Author YS shared all authors during research conduction. All authors shared in the preparation of the manuscript. All authors read and approved the final manuscript.

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