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Effect of grit-blasting air pressure on adhesion strength of resin to titanium

Aftab Ahmed Khan [a,*], Abdulaziz A. Al Kheraif [a], Saleh Mohammed Alhijji [b], Jukka Pekka Matinlinna [c,d]

[a] Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh 11433, Saudi Arabia
[b] Dental Health Department, College of Applied Medical Sciences, King Saud University, Riyadh 11433, Saudi Arabia
[c] Dental Materials Science, Faculty of Dentistry, The University of Hong Kong, Hong Kong, China
[d] Visiting Professor Program, King Saud University, Riyadh, Saudi Arabia

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ABSTRACT

Aim: The objective of this laboratory study was to analyze the effect of different grit-blasting pressures on adhesion of resin to silica-coated and silanized Ti using the shear bond test.
Materials and methods: A total of 24 commercially pure grade 2 Ti coupons (1 mm × 20 mm × 40 mm) were prepared and randomly assigned to 4 groups based on surface treatment: 150 kPa grit-blasting pressure with Rocatec™ Soft (group 1) for 10 s. Similarly, groups 2, 3 and 4 were treated at 280 kPa (control), 330 kPa and 380 kPa grit-blasting pressures, respectively, and followed by silanization. A total of 10 resin stubs per group were bonded onto each treated surface with photopolymerization. The shear bond strength was measured after 24 h dry storage in a desiccator, 2 months H_2O storage, and 4 months H_2O storage. Data were analyzed using descriptive statistics and two-way ANOVA ($p < 0.05$).
Results: After 24 h, initial SBS values of tested groups were significantly higher (32.0% for group 1, 39.1% for group 3, and 23.9% for group 4) than the control (group 2). After artificial aging, SBS values decreased in all the groups. The highest adhesion strength was seen in 150 kPa (13.0 ± 3.0) and 280 kPa (4.9 ± 2.4) after 2 months, and 4 months artificial aging, respectively.
Conclusion: A lower grit-blasting pressure might promote adhesion strength in long term water aging.

1. Introduction

With the advent of advanced casting machines the use of titanium and titanium alloys has increased dramatically in dentistry [1]. Titanium is being frequently used for crowns, dental implants, porcelain fused to metal (PFM) crowns, and as a frame work for CAD/CAM-milled fixed partial dentures [2]. Titanium and its alloys have excellent biocompatibility, high strength, low density, and high corrosion resistance. In addition, veneering porcelain can be fused and bonded to titanium surface in PFM restorations [3]. The popularity of using clinically titanium as a prosthetic restoration material has gained researchers' continuous interest in enhancing its adhesion strength with resin composite cements.

Several methods have been employed to promote the adhesion strength of titanium with resins. Among them, tribochemical silica-coating (Rocatec™) is probably the most widely used [4]. This technique comprises silica modified grit-blasting particles followed by the application of a silane coupling agent. This process is called silanization [1]. Silanes which are bifunctional molecules help in forming a chemical bond between dissimilar materials with a silica layer on the titanium surface after silica-coating [5]. Silanization aims to provide increased surface free energy to improve surface wettability of the adhesive [6]. 3-Methacryloxyproyltrimethoxysilane (MPS) is the frequently used active silane monomer in commercially available dental silane primers [7], considered a “gold standard” for adhesion promotion between resins and silica coated metals [8]. However, studies on the effect of artificial aging *i.e.*, thermo-cycling, water storage *etc.* on the predictability of long term adhesion (bond strength) are still needed [4].

On the other hand, the operating air pressure is a very important factor. The effect of grit-blasting pressure has been evaluated on resin to zirconia bonding. Heikkinen et al. suggested that higher adhesion strength of resin to zirconia was possible using a higher tribochemical operating pressure [9]. One can postulate that due to higher kinetic energy of the grit particles, higher surface roughness with increased embedding rate of the silica

* Corresponding author. Tel.: +966 59 1125997.
E-mail addresses: aakjk@hotmail.com (A.A. Khan), aalkhuraif@ksu.edu.sa (A.A. Al Kheraif), smalhijji@ksu.edu.sa (S.M. Alhijji), jpmat@hku.hk (J.P. Matinlinna).

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Table 1
Mean surface roughness (S_a, μm) and standard deviation.

Grit-blasting pressure (kPa)		No treatment	150	280	330	380
Surface roughness (mean ± SD)	**Without Sil**	0.52 ± 0.04	0.57 ± 0.09	0.64 ± 0.12	0.68 ± 0.10	0.76 ± 0.16
	With Sil		0.60 ± 0.08	0.66 ± 0.09	0.68 ± 0.11	0.75 ± 0.12

Key: S_a = arithmetic average of the 3D roughness, Sil = silane primer used.

Table 2
Mean and standard deviation values of SBS with percentage of enhancement.

Storage condition	Grit-blasting pressure (kPa)	Mean ± SD (MPa)	Change in SBS (%)
24 h dry storage in a desiccator	150	$13.1 \pm 1.8_a^A$	47.1
	280 (control)	8.9 ± 2.0^B	0
	330	$14.6 \pm 2.8_a$	64.0
	380	$11.7 \pm 1.3_a$	31.4
2 m storage in distilled water	150	$13.0 \pm 3.0_b^A$	88.4
	280 (control)	$6.9 \pm 2.6_c^{B,C}$	0
	330	$8.5 \pm 3.9_{b,c}$	23.1
	380	$7.7 \pm 2.2_c$	11.5
4 m storage in distilled water	150	$3.0 \pm 1.1_d$	−38.7
	280 (control)	$4.9 \pm 2.4_d^C$	0
	330	$3.3 \pm 1.7_d$	−32.6
	380	$2.5 \pm 1.2_d$	−48.9

Key: SBS = shear bond strength.
Different superscript uppercase letters demonstrate insignificant differences between the aging groups. Different subscript lowercase letters demonstrate the insignificant differences between the grit-blasting air pressure groups.

Table 3
Failure mode analysis after different aging conditions

Aging method	Operating pressure (kPa)											
	150			280			330			380		
	A (%)	M	C	A (%)	M	C	A (%)	M	C	A (%)	M	C
24 h dry storage	80	20	0	100	0	0	60	40	0	100	0	0
2 months storage in distilled water	80	20	0	100	0	0	100	0	0	100	0	0
4 months storage in distilled water	100	0	0	100	0	0	100	0	0	100	0	0

Key: A = adhesive failure, M = mixed failure, C = cohesive failure.

particles take place. This increased surface area helps in forming a stable bond at the interface of resin and zirconia [10]. Or, this is probably due to the higher number of particles blasted per unit time caused the silica particles to embed on the substrate with a higher operating air pressure [11]. However, sintered zirconia is much harder material than Ti. The effect of grit-blasting air pressure is still need to be explored on Ti substrate to find out the missing optimal parameters for durable resin Ti bonding. Adhesion strength testing by using shear bond strength test (SBS) is today disputed [12,13]. A novel approach could be the strain energy release rate by which adhesion of different systems can be evaluated [14]. However, SBS may give a relatively reliable and quick assessment of adhesion [1,2]. The tensile strength test on such resin Ti adhesion specimens is very cumbersome and tedious to perform.

Several studies have been conducted to analyze the effect of long term water storage at the interface of resin–metal on bond strength [1,15]. In these studies reduced bond strength values were observed. Nonetheless, there are no studies conducted to analyze the effect of different grit-blasting pressures on Ti surface using silanization and *bis*-GMA-based resins. Despite the theories related to the optimal pressure, distance and angle, there are no published data on varying grit-blasting pressures [16]. Some new techniques are being recently proposed for optimal bonding. A research by Ho et al. highlighted the importance of the distance and angle on grit-blasting procedures on both ZrO_2 and Ti [15]. On the other hand, Kern et al. proposed a low-pressure air-abrasion approach, in which their parameters used were 0.05 MPa/0.5 bar [17]. The current laboratory study aimed to evaluate effect of different grit-blasting pressures on adhesion (shear bond) strength of resin with Ti. The Rocatec™ system was preferred over Cojet™ Sand because the former demonstrates higher SBS values [18]. The null hypothesis tested was that a *bis*-GMA resin composite provides similar bond strength with different grit-blasting pressures in all aging methods used.

2. Materials and methods

Commercially pure grade 2 Ti sheet (Permascand, Ljungaverk, Sweden; > 99%) was used. With the help of a saw blade, the Ti sheets were cut into smaller coupons with the final dimension of 1 mm × 20 mm × 40 mm. A total of 24 coupons were cut. They were randomly divided into 4 sub-groups.

2.1. Surface treatment of coupons

The upper halves of the surface of the coupons were grit-blasted with the Rocatec™ Soft (3M ESPE, Seefeld, Germany) powder for tribochemical silica-coating. The treatment for group 1 group was carried out with a slowly rotating constant motion, in a jet at 150 kPa from a perpendicular distance of 10 mm to the titanium surface for 10 s (the manufacturer's recommendation). Similarly, same treatment method was followed for groups 2 (control), 3 and 4, but at 280 kPa *i.e.,* control, 330 kPa, and 380 kPa grit-blasting pressures, respectively. The treated coupons were ultrasonically cleansed in 70% acetone for 10 min and left for air dry.

2.2. Primer and resin bonding

A commercially available dental silane coupling agent, ESPE™ Sil (3M ESPE, Seefeld, Germany) was applied on the silica-coated Ti substrate and left for 5 min for air dry. Before the bonding process, a transparent polyethylene mold with an inner diameter of 3.6 mm and 3.5 mm of height was kept and pressed on the specimen surface manually. A light cured *bis*-GMA-based filled adhesive resin composite cement Stickflow™ (Stick Tech Ltd., Turku, Finland) was used according to the manufacturer's instruction and filled into the mold. The curing procedure was performed for 40 s from the top of the mold and then from the lateral side for 40 s by using a light curing unit (Elipar™ 2500, 3M ESPE, Minneapolis, USA). The molds were removed with a great care after curing by pressing the stub with a hand instrument.