International Journal of Adhesion & Adhesives 65 (2016) 41–46

Contents lists available at ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

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

ARTICLE INFO

Article history:
Accepted 11 November 2015
Available online 28 November 2015

Keywords:
Adhesion
Grit-blasting pressure
Resin
Silane primer
Titanium

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).

http://dx.doi.org/10.1016/j.ijadhadh.2015.11.003
0143-7496/

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 (%)
	150	$13.1 \pm 1.8_a^A$	47.1
24 h dry storage in a desiccator	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
	150	$13.0 \pm 3.0_b^A$	88.4
2 m storage in distilled water	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
	150	$3.0 \pm 1.1_d$	−38.7
4 m storage in distilled water	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.

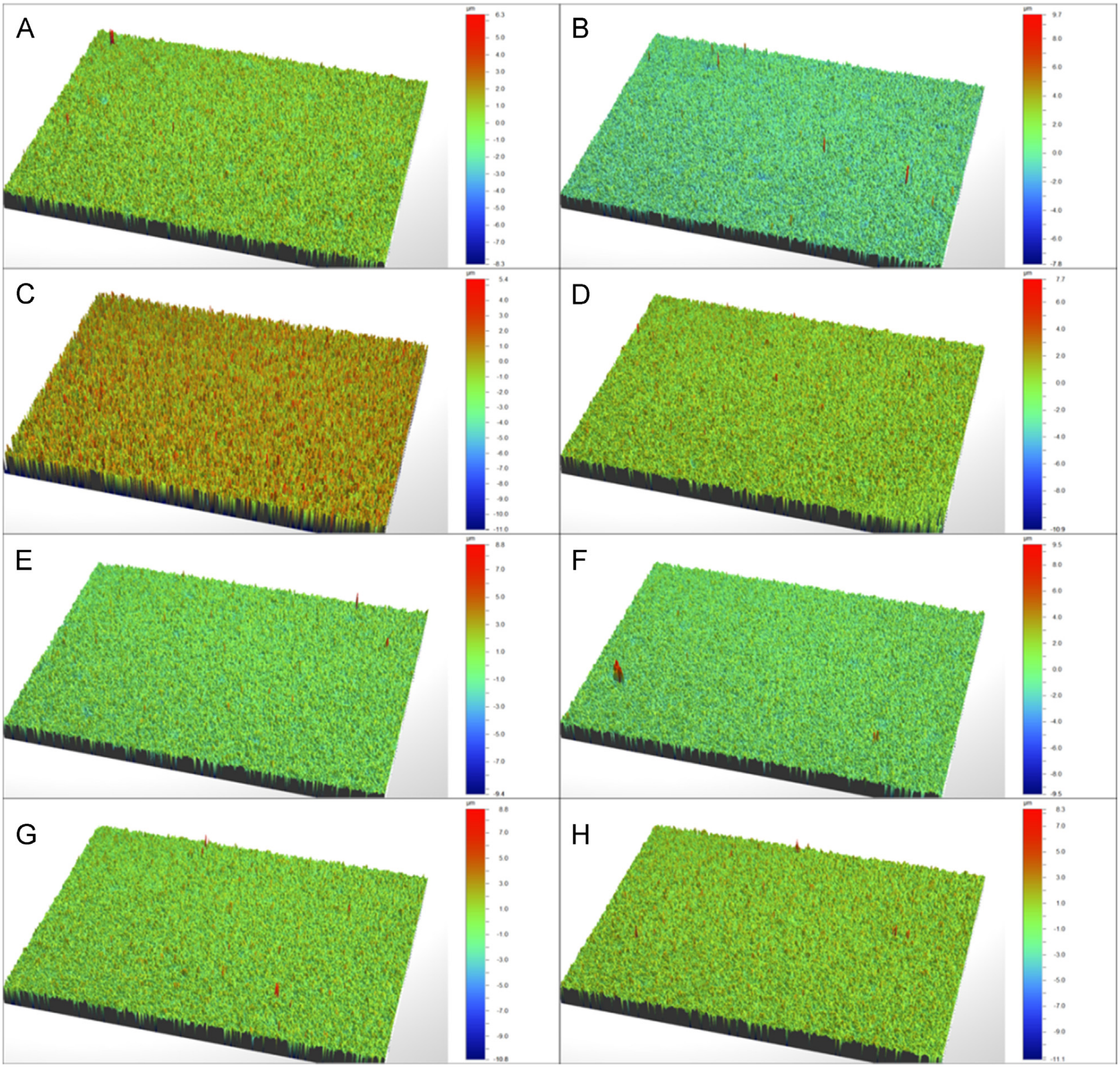

Fig. 1. The 3D surface roughness profile images A and B: grit-blasting at 150 kPa and grit-blasting at 150 kPa + application of silane primer respectively. C and D: grit-blasting at 280 kPa and grit-blasting at 280 kPa + application of silane primer respectively. E and F: grit-blasting at 330 kPa and grit-blasting at 330 kPa + application of silane primer respectively. G and H: grit-blasting at 380 kPa and grit-blasting at 380 kPa + application of silane primer respectively.

On each Ti coupon, 5 resin stubs were photo-polymerized. Samples from each group were placed in distilled water for 2 months and 4 months periods for artificial aging. The baseline groups (24 h dry storage in a desiccator) were tested from each study group.

2.3. Surface roughness analysis

Surface roughness of all specimens was analyzed with a 3D optical non-contact surface profiler (UMT 1 Bruker, Campbell, CA, USA). The surface roughness from each group was measured before and after the surface treatment. The selected parameter to analyze the amplitude properties of the surface was S_a (the so-called roughness average). Based on non-contact scanning white light interferometry with the objective standard camera of magnification 5 ×, the machine was placed on a vibration isolation table. The profile meter scanned all sample areas approximately 1.3 mm × 1.0 mm. The scanning area was situated at the center part of the surface. Vision64 (v 5.30) application software (Bruker, Campbell, CA, USA) was used to control the precision and the measurements of surface roughness parameters.

2.4. Adhesion strength test

Shear bond strength was measured in a universal testing machine (Model no. 3369 Instron, USA) with a cross-head speed of 1.0 mm/min. The constant loading was applied until failure of each specimen occurred. A custom made specimen holder was used for Ti coupons during testing. The shear bond strength (SBS) was calculated according to the following formula:

$$\text{SBS} = \text{fracture load}/\text{area of bonded resin stub}$$

2.5. Failure mode analysis

Failure mode analysis was examined with an optical microscope (Nikon SMZ 1000, Tokyo; Japan). Adhesive, mixed or cohesive failure modes were assigned according to proportion of resin composite cement remained on the Ti coupon. When $\leq 33\%$ resin cement remained on Ti substrate, it was considered adhesive failure; when $\geq 34\%$ but $\leq 66\%$, mixed failure mode, whereas $\geq 67\%$ and $\leq 100\%$ was considered cohesive failure [19].

2.6. Statistical analysis

The SPSS version 21.0 (Statistical Package for Statistical Science, Chicago, IL, USA) was used to analyze the results. The level of statistical significance p was set as 0.05 in all the tests. A two-way factorial analysis of variance (ANOVA) followed by Tukey's *post-hoc* tests was carried out to explore if there was any statistical correlation between the individual groups.

3. Results

Table 1 presents the surface roughness (S_a, µm) values of different surface treatment methods. The highest roughness value was observed in 380 kPa group (0.76 ± 0.16), whereas the lowest roughness was found in Ti substrate without any treatment (0.52 ± 0.04).

The descriptive statistics of the shear bond strength are presented in Table 2. According to the results of the two-way analysis of variance (ANOVA), a statistically significant difference was observed between the experimental groups and the control group. Irrespective of the grit-blasting air pressure, the shear bond strength values decreased with the aging time in the control and all the experimental groups. Among the 2 months aging groups, a significant difference was observed only between 150 kPa and 280 kPa. In addition, no significant difference was observed among the groups on one variable, *i.e.*, grit-blasting pressure at the end of 4 months of aging as seen in Fig. 2.

Table 3 presents the distribution of failure modes after the shear bond test. In the 24 h study groups all failures were adhesive except 150 kPa and 330 kPa groups, where 20% and 40% were mixed failures, respectively. In the 2 months study groups, only 20% of the specimens showed mixed failure mode whereas a complete adhesive failure mode was observed in the 4 months study groups.

4. Discussion

The null hypothesis could not be verified in this study. There were significant differences between grit-blasting pressures and adhesion strength.

There is an increased demand for the longevity of a restoration in the oral cavity and the key concept of attaining strong and resistant bonding of resin to Ti is grit-blasting followed by a silane coupling agent [19]. Grit-blasting (sandblasting) is a common and frequently used method in dental procedures that counts on kinetic energy theory [16] *i.e.*, kinetic energy$=½mv^2$. The rationale of discovering the optimal pressure is behind this simple basic science theory, and to analyze the effect of speed of grit particles in creating a micro-mechanical retention for enhanced bonding of resin to Ti.

As can be seen in Table 1, the surface roughness had a direct correlation with the grit-blasting pressure. Considering the grit-blasting pressure, silicatized particles of Rocatec™ Soft produced more micro-roughness at 380 kPa (0.76 ± 0.16). This could be attributed to the bombardment of silica-coated alumina particles at a higher speed. The effect of different blasting pressures can also be observed by evaluating the 3D surface roughness profile images seen in Fig. 1.

The grit-blasting was carried out using 30 µm silica-coated alumina particles. Such smaller particles (powder) are used in chair-side repair of certain restorations in dentistry [5]. We didn't use the powder (particle size 110 µm) that is widely used in dental laboratories. It was contemplated that the smaller grit particles would not affect the surface integrity of the substrate but provide a homogeneous coating. Microcracks created by the bigger grit particles might be a source of stress concentration [20], thereby weakening the material. The surface roughness created by the blasting pressure played a significant role in adhesion strength of resin to Ti. However, this was not studied in the current study.

All the baseline results of adhesion testing were obtained after storage in a desiccator only and these results yielded the highest adhesion strength with the lowest standard deviation values. After 2 months of water aging, adhesion strength values started decreasing with increasing standard deviation values, and after 4 months of water aging the standard deviation values were at the level of 30% to 50% of the mean values (Table 2). This might be explained by the hydrolytic effect of the storage medium. Over the time H_2O penetration at the interface of resin to silica-coated Ti will take place and adsorption of H_2O by siloxane bonds, which are susceptible for hydrolytic degradation in a silane generated adhesion film and may weaken the adhesion strength [8,21].

In this study, water storage over 4 months perhaps yielded the most surprising results. Regardless of the operating pressure, the results obtained for adhesion strength were all below 5 MPa which is a minimum requirement set by an ISO standard [22]. Such results might suggest that this type of composite cement is not ideal for titanium application (Fig. 2). This Stickflow™ was selected because it is a widely used flowable composite with *bis*-GMA for cementation of indirect fiber-reinforced composite restorations.

Elemental analysis of grit-blasted or grit-blasted and silanized surfaces was not carried out in this study. Some previous studies [2,8,11] suggest that Ti surface after silica-coating has varying concentration of Si and O_2. The lower adhesion strength values could be attributed to a lower silica concentration on Ti substrate that Rocatec™ Soft treatment might not have fully covered. Secondly, a possible uneven distribution of Si might have become the

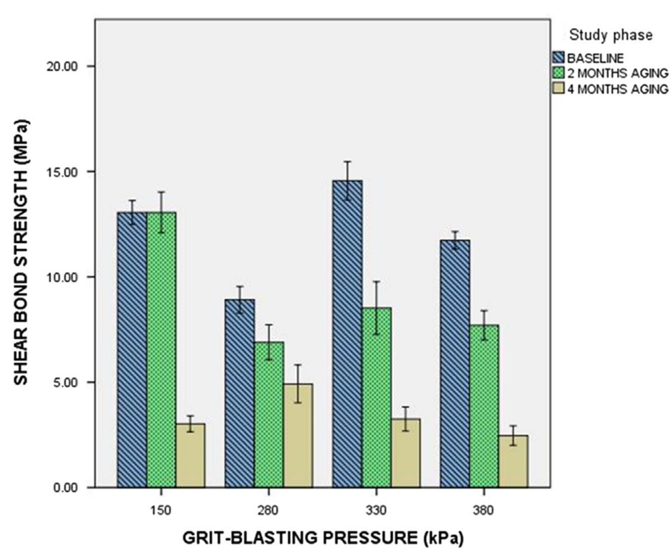

Fig. 2. Comparisons on mean adhesion (shear bond) strengths with different grit-blasting pressures.

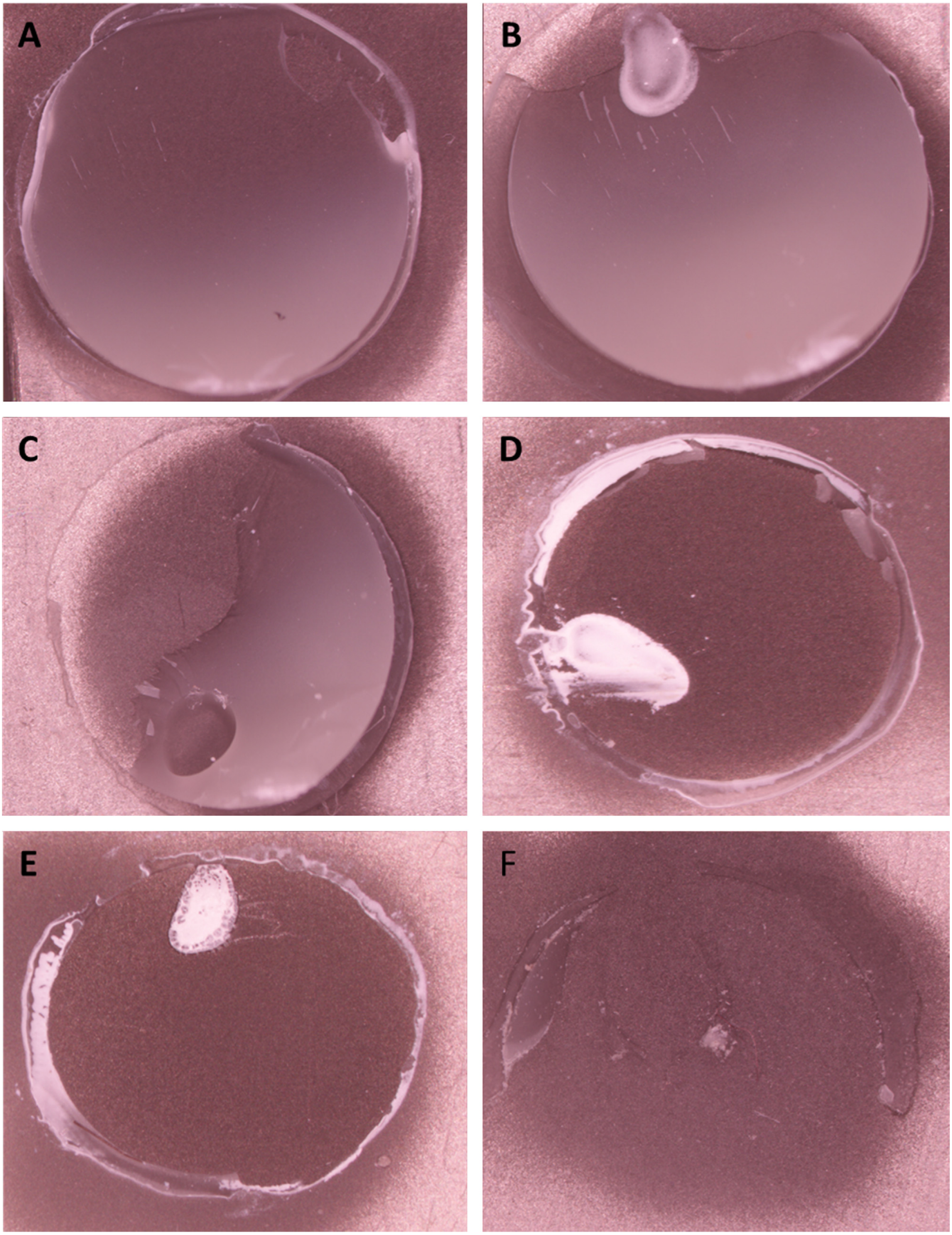

Fig. 3. Optical microscope images A and B: mixed failure mode in 150 kPa and 330 kPa (24 h) specimens, respectively. C and D: adhesive failure in 330 kPa and 380 kPa (2 m) specimens, respectively. E and F: adhesive failure in 280 kPa and 380 kPa (4 m) specimens, respectively.

reason of high standard deviations in SBS values [2]. The working parameters on Ti surfaces were strongly followed according to the manufacturer's recommendation and thereby, to avoid handling errors. The required operations were performed by the same operator.

It was not surprising that most of the failure modes were adhesive in nature when qualitative microscopic analysis was performed. The failure mode analysis may vary between the studies. Hence, the idea of mixed failure might not always be supported [23]. In the baseline study groups, few of the mixed failure modes were observed. However, in general, H_2O aging had a detrimental effect on the adhesive strength. Apart from the 150 kPa group in 2 months of aging where 20% of the specimens showed a mixed failure mode, the rest of all the groups exhibited the adhesive failure mode (Table 3). After the 4 months aging, the adhesion strength dropped to such a low level that none of the specimens showed any mixed or cohesive failure mode (Fig. 3). In the near future, studies on lowering the operational air pressure might be important to carry out in order to clarify the ambiguity.

5. Conclusion

As conclusions we might summarize that:

- Water aging significantly decreased the adhesion strength regardless of the surface treatment used.
- With resin bonding, lowering the recommended operational air pressure might promote strong adhesion.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgment

We extend sincere appreciation to the Deanship of Scientific Research, King Saud University for funding/supporting this research through Research Group no.—IRG 14-31.

References

[1] Ozcan M, Valandro LF. Bond strength of two resin cements to titanium after different surface conditioning methods. Gen Dent 2012;60(1):e6–12.
[2] Matinlinna JP, Lassila LV, Vallittu PK. The effect of a novel silane blend system on resin bond strength to silica-coated Ti substrate. J Dent 2006;34(7):436–43.
[3] Haag P, Nilner K. Bonding between titanium and dental porcelain: a systematic review. Acta Odontol Scand 2010;68(3):154–64.
[4] Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. Dent Mater 2012;28(5):467–77.
[5] Ozcan M. The use of chairside silica coating for different dental applications: a clinical report. J Prosthet Dent 2002;87(5):469–72.
[6] Ural C, Külünk T, Külünk S, Kurt M, Baba S. Determination of resin bond strength to zirconia ceramic surface using different primers. Acta Odontol Scand 2011;69(1):48–53.
[7] Aboushelib MN, Matinlinna JP, Salameh Z, Ounsi H. Innovations in bonding to zirconia-based materials: Part I. Dent Mater 2008;24(9):1268–72.
[8] Matinlinna JP, Tsoi JK, de Vries J, Busscher HJ. Characterizations of novel silane coatings on titanium implant surfaces. Clin Oral Implant Res 2013;24(6):688–97.
[9] Heikkinen TT, Lassila LV, Matinlinna JP, Vallittu PK. Effect of operating air pressure on tribochemical silica-coating. Acta Odontol Scand 2007;65(4):241–8.
[10] Darvell BW. Materials science for dentistry. 6th ed.. Hong Kong: Woodhead; 2000. p. 394–6.
[11] Matinlinna JP, Lassila LVJ, Kangasniemi I, Vallittu PK. Isocyanato- and methacryloxy-silanes promote bis-GMA adhesion to titanium. J Dent Res 2005;84(4):360–4.
[12] Söderholm KJ. Critical evaluation of adhesive test methods used in dentistry. J Adhes Sci Technol 2009;23(7–8):973–90.
[13] Darvell BW. Adhesion strength testing – time to fail or a waste of time? J Adhes Sci Technol 2009;23(7–8):935–44.
[14] Elsaka SE. Effect of surface pretreatments on the bonding strength and durability of self-adhesive resin cements to machined titanium. J Prosthet Dent 2013;109(2):113–20.
[15] Taira Y, Yanagida H, Matsumura H, Yoshida K, Atsuta M, Suzuki S. Adhesive bonding of titanium with a thione-phosphate dual functional primer and self-curing luting agents. Eur J Oral Sci 2000;108(5):456–60.
[16] Ho BJ, Tsoi JKH, Liu D, Lung CYK, Wong HM, Matinlinna JP. Effects of sandblasting distance and angles on resin cement bonding to zirconia and titanium. Int J Adhes Adhes 2015;62:25–31.
[17] Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. J Dent Res 2009;88(9):817–22.
[18] Abi-Rached Fde O, Fonseca RG, Haneda IG, de Almeida-Júnior AA, Adabo GL. The effect of different surface treatments on the shear bond strength of luting cements to titanium. J Prosthet Dent 2012;108(6):370–6.
[19] Lung CY, Liu D, Matinlinna JP. Surface treatment of titanium by a polydimethylsiloxane coating on bond strength of resin to titanium. J Mech Behav Biomed Mater 2015;41:168–76.
[20] Paranhos MP, Burnett Jr LH, Magne P. Effect of Nd:YAG laser and CO_2 laser treatment on the resin bond strength to zirconia ceramic. Quintessence Int 2011;42(1):79–89.
[21] Khan AA, Siddiqui AZ, Al-Kheraif AA, Zahid A, Divakar DD. Effect of different pH solvents on micro-hardness and surface topography of dental nano-composite: an in vitro analysis. Pak J Med Sci 2015;31(4):854–9.
[22] Liu D, Pow EHN, Tsoi JKH, Matinlinna JP. Evaluation of four surface coating treatments for resin to zirconia bonding. J Mech Behav Biomed 2014;32:300–9.
[23] Della-Bona A. Important aspects of bonding resin to dental ceramics. J Adhes Sci Technol 2009;23(7–8):1163–76.