Effect of alumina air-abrasion on mechanical bonding between an acrylic resin and casting alloys

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(Received 24 December 2008 and accepted 15 January 2009)

Abstract: This study examined the effect of alumina air-abrasion with different pressure on bonding between an acrylic resin and casting alloys. Disk specimens (8 and 10 mm in diameter) were cast from a silver-palladium-copper-gold (Ag-Pd-Cu-Au, Castwell M.C.12) alloy and a titanium-aluminum-niobium alloy (Ti-6Al-7Nb, T-Alloy Tough). The disks were air-abraded with alumina particles (50-70 μ m) under different air-pressures (0 unabraded, 0.1, and 0.6 MPa). The disk pairs were bonded together with a tri-*n*-butylborane (TBB)-initiated acrylic resin, and shear bond strengths were determined both before and after thermocycling. Bond strength varied from a maximum of 37.1 MPa to a minimum of 3.6 MPa for the Ag-Pd-Cu-Au alloy, whereas bond strength to Ti-6Al-7Nb alloy ranged from 34.7 MPa to 0.1 MPa. Specimens abraded with 0.6 MPa pressure recorded the greatest post-thermocycling bond strength (21.7 MPa and 17.9 MPa), and unabraded specimens showed the lowest strength (3.6 MPa and 0.1 MPa) for both alloys. Post-thermocycling bond strength to the Ag-Pd-Cu-Au alloy was higher than that to the Ti-6Al-7Nb alloy under identical air-abrading conditions. It can be concluded that alumina air-abrasion with an air-pressure of 0.6

MPa is effective in enhancing retentive characteristics of the TBB-initiated resin joined to the alloys. (J Oral Sci 51, 161-166, 2009)

Keywords: abrasion; Ag-Pd-Cu-Au alloy; alumina; bonding; Ti-6Al-7Nb alloy; resin.

Introduction

The development of laboratory and adhesive techniques has led to the application of silver-palladium-copper-gold (Ag-Pd-Cu-Au) alloy, titanium-aluminum-niobium (Ti-6Al-7Nb) alloy, and other alloys for resin-bonded fixed partial dentures (RBFDPs) (1-6). The bonding between polymeric materials and Ag-Pd-Cu-Au alloys has improved by use of thiol or thione monomers (7,8), whereas bonding to Ti-6Al-7Nb has improved through the use of primers with phosphate (9,10).

Before the bonding procedure, the priming systems require air-abrasion with alumina to mechanically clean the surfaces and to increase the surface bonding area. It is therefore necessary to determine the appropriate conditions for alumina air-abrasion. Although the effects of alumina abrasion on bond strength have been reported for cobalt-chromium, nickel-chromium, and gold alloys as well as titanium (11-15), only limited information is available about Ag-Pd-Cu-Au and Ti-6Al-7Nb alloys (16). The purpose of the current study was to evaluate the influence of air-abrasion with alumina on mechanical bonding between an acrylic resin and two casting alloys.

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Table 1 Materials assessed

Material / Trade name	Abbreviation	Manufacturer	Lot number	Composition (mass %)
Casting alloy				
Castwell M.C.12	Ag-Pd-Cu-Au	GC Corp.	0602021 0707201	46 Ag, 20 Pd, 20 Cu, 12 Au
T-Alloy Tough	Ti-6Al-7Nb	GC Corp.	408041	86.5 Ti, 7 Nb, 6 Al
Alumina powder				
Hi-Aluminas		Shofu Inc.		50-70 μm grain sized alumina
Luting material				
Methyl methacrylate	MMA	Wako Pure Chemical Ind., Ltd.	ALP5224	99.8 MMA
Super-Bond Catalyst		Sun Medical Co., Ltd.	RT13	TBB
Super-Bond Opaque powder		Sun Medical Co., Ltd.	SF1	PolyMMA, Titanium oxide

MMA, Methyl methacrylate; TBB, Tri-*n*-butylborane derivative

Materials and Methods

An Ag-Pd-Cu-Au alloy (Castwell M.C.12, GC Corp., Tokyo, Japan) and Ti-6Al-7Nb alloy (T-Alloy Tough, GC Corp.) designed for cast restorations and partial denture frameworks were selected as the substrate materials. Alumina powder (50 – 70 μm grain size; Hi-Aluminas, Shofu, Inc., Kyoto, Japan) was used for air-abrasion. An unfilled acrylic resin material initiated with tri-*n*-butylborane derivative (TBB) was employed as the luting material. The details of the materials used in the experiment are summarized in Table 1.

A total of 66 paired disk specimens (8 and 10 mm in diameter; 2.5 mm in thickness) were prepared from the two alloys, respectively. The Ag-Pd-Cu-Au alloy was cast in a cristobalite investment material (Cristoquick 20, GC Corp.) using a high-frequency argon-arc casting machine (Argoncaster, Shofu, Inc.), whereas the Ti-6Al-7Nb alloy was cast in a magnesia-based investment material (Selevest CB, Selec, Osaka, Japan) using a spin-cast centrifugal apparatus (Ticast Super R, Selec). All disks were sanded with 800-grit silicon-carbide abrasive paper.

Sixty-six disk pairs were divided into three sets of 22 disk pairs each. Of these, the first set (22 pairs for each alloy) was not air-abraded with alumina, and considered as the control group. The second set (22 pairs for each alloy) was air-abraded with alumina by means of an air-borne particle abrader (Jet Blast II, J. Morita Corp., Suita, Japan) for 10 s with 0.1 MPa air-pressure. The remaining set was also air-abraded with 0.6 MPa air-pressure. The distance of the orifice from the disk surface was 10 mm. After surface preparation, a piece of tape with a circular hole, 5 mm in diameter, was positioned on each 10-mm disk specimen to define the bond area. The identically treated disk pairs

were bonded together with the TBB-initiated non-adhesive resin using the brush-dip technique. Twenty-two specimens were prepared for each surface preparation group. Thirty minutes after preparation, the specimens were immersed in 37°C water for 24 h before testing for bond strength. One-half of the specimens (11 specimens; two alloys; three surfaces) were subsequently thermocycled in water between 5°C and 55°C for 10,000 cycles with 1-minute dwell time per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co., Ltd., Tokyo, Japan).

Each specimen was embedded in an acrylic resin mould and seated in a shear-testing jig. Shear bond strengths were then determined with a mechanical testing machine (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/minute. Debonded surfaces were observed through an optical microscope (8x; SZX9, Olympus Corp., Tokyo, Japan), and the failure modes were classified into the following three categories: adhesive failure, cohesive failure within the luting material, and combination of adhesive and cohesive failures. Typical specimens after surface preparation, and before bonding, were observed with a scanning electron microscope (S-4300, Hitachi High-Technologies Corp., Tokyo, Japan) with an accelerating voltage of 15 kV.

The bond strength results were analyzed with software for statistical analysis (SPSS version 15.0, SPSS, Inc., Chicago, IL, USA). Equality of variance of the bond strengths was primarily analyzed by Levene test and F test. When the Levene test and F test did not show equality of variances, Steel-Dwass comparison (Kypplot 4.0, KyensLab, Tokyo, Japan) was performed to evaluate the effect of alumina air-abrasion. Mann-Whitney *U*-test was used to evaluate the difference in bond strengths between pre-

Table 2 Results of shear bond strength in MPa

Alloy	Air-abrasion	0 thermocycles				10,000 thermocycles					Reduction from 0 to 10,000**
		Median	Mean	SD	Category*	Median	Mean	SD	PTF	Category*	
Ag-Pd-Cu-Au	None	18.7	17.8	2.8	a	3.2	3.6	1.3	0	c	Significant
Ag-Pd-Cu-Au	0.1 MPa	36.4	36.2	2.1	b	15.3	15.9	1.7	0	d	Significant
Ag-Pd-Cu-Au	0.6 MPa	37.1	37.1	1.4	b	22.4	21.7	1.5	0	e	Significant
Ti-6Al-7Nb	None	12.3	11.9	1.6	f	0.0	0.1	0.1	5	h	Significant
Ti-6Al-7Nb	0.1 MPa	35.0	34.7	2.1	g	10.4	10.3	2.2	0	i	Significant
Ti-6Al-7Nb	0.6 MPa	34.5	34.5	1.9	g	17.5	17.9	1.1	0	j	Significant

$n = 11$; SD, Standard deviation; PTF, Number of pre-testing failures; *Identical letters indicate that the values are not significantly different (Steel-Dwass comparison; $P > 0.05$). **Significant difference between pre- and post-thermocycling bond strengths was found (Mann-Whitney U -test; $P < 0.05$).

Table 3 Failure modes after shear bond testing

Alloy	Air abrasion	0 thermocycle			10,000 thermocycles		
		A	AC	C	A	AC	C
Ag-Pd-Cu-Au	None	11	0	0	11	0	0
Ag-Pd-Cu-Au	0.1 MPa	1	10	0	0	11	0
Ag-Pd-Cu-Au	0.6 MPa	2	9	0	1	10	0
Ti-6Al-7Nb	None	11	0	0	11	0	0
Ti-6Al-7Nb	0.1 MPa	1	10	0	5	6	0
Ti-6Al-7Nb	0.6 MPa	3	8	0	1	10	0

A, Adhesive failure at the resin-alloy interface; C, Cohesive failure within the resin; AC, Combination of adhesive and cohesive failures

and post-thermocycling groups, or the difference in bond strengths between the two alloys under identical surface preparation. Significance level was set at 0.05 for all analyses.

Results

Levene test and F test run on the bond strength results revealed that several groups did not show equality of variance. Steel-Dwass test was therefore applied to evaluate the influence of alumina air-abrasion on bond strength. In addition, Mann-Whitney U -test was used for comparison of bond strengths between two different conditions.

Table 2 shows the results of shear bond testing and statistically categorized groupings. Bond strength varied from a maximum of 37.1 MPa to a minimum of 3.6 MPa for the Ag-Pd-Cu-Au alloy. Bond strength to Ti-6Al-7Nb

alloy ranged from 34.7 MPa to 0.1 MPa. Unabraded specimens showed lower bond strength (categories a, f, c, and h) than the air-abraded specimens for both alloys. Among the three abrasion conditions, the specimens abraded with 0.6 MPa pressure recorded the greatest post-thermocycling bond strength (21.7 MPa for the Ag-Pd-Au-Cu alloy and 17.9 MPa for the Ti-6Al-7Nb alloy), and unabraded specimens showed the lowest strength (3.6 MPa for the Ag-Pd-Au-Cu alloy and 0.1 MPa for the Ti-6Al-7Nb alloy). Post-thermocycling bond strength to the Ag-Pd-Cu-Au alloy was significantly higher than that to the Ti-6Al-7Nb alloy under an identical air-abrading condition ($3.6 > 0.1$, $15.9 > 10.3$, and $21.7 > 17.9$). Bond strength of all six groups was reduced by thermocycling.

Table 3 shows the mode of failures after shear bond testing. All of the unabraded specimens showed adhesive

failure, whereas many alumina-blasted specimens showed a combination of adhesive and cohesive failures. Complete cohesive failure was not observed in any of the specimens in the current study.

Figure 1 shows the ground surfaces of two alloys. Scratches generated by the abrasive paper could be seen

in both specimens. Figures 2 and 3 show the alloy surfaces after air-abrasion. The surfaces of the specimens became rougher in accordance with air-pressure. The Ag-Pd-Cu-Au alloy surface abraded with 0.6 MPa pressure was rougher than the Ti-6Al-7Nb alloy surface abraded with the same pressure.

Fig. 1 Scanning electron micrographs of (a) Ag-Pd-Cu-Au alloy and (b) Ti-6Al-7Nb alloy ground with #800 SiC abrasive paper (original magnification, $\times 500$).

Fig. 2 Scanning electron micrographs of alumina-abraded Ag-Pd-Cu-Au alloy with 0.1 MPa (a) and 0.6 MPa (b) air-pressures (original magnification, $\times 500$).

Fig. 3 Scanning electron micrographs of alumina-abraded Ti-6Al-7Nb alloy with 0.1 MPa (a) and 0.6 MPa (b) air-pressures (original magnification, $\times 500$).

Discussion

This study compared the influence of different alumina-abrasion pressures on bonding between an acrylic resin and two casting alloys. The authors did not use adhesive functional monomers in order to evaluate the single effect of alumina air-abrasion.

As shown in Table 1, alumina air-abrasion effectively enhanced bond strength of the resin material (categories a and b; f and g). The difference between the two air-pressure conditions, however, was not significant (categories b or g) at the pre-thermocycling state. This is probably due to cohesion of resin material to the roughened alloys before penetration of water into the adhesive interface. Also, the result was slightly different from that of a previous paper reporting effects of alumina air-abrasion on bond strength to Ti-6Al-7Nb alloy (16). The authors speculate that the disparity in results could be attributed to the differences in the bonded material, the distance between the surface to be abraded and aperture of the air-borne particle abrader (10 mm in this paper and 20 mm in reference 16), and the abrasion time period (10 s in this paper and 15 s in reference 16).

After thermocycling, significant differences in bond strength were found among the three surface conditions. Specifically, specimens abraded with 0.6 MPa pressure recorded the highest bond strength (categories e and j), while unabraded specimens resulted in the lowest bond strength (categories c and h). The results can be explained by the difference in surface roughness of the specimens shown in Figs. 1 – 3. The rougher surface of the Ag-Pd-Cu-Au alloy as compared with the Ti-6Al-7Nb alloy may be due to the difference in hardness between the two alloys (146 VHN for Ag-Pd-Cu-Au alloy and 320 VHN for Ti-6Al-7Nb alloy). Although the rougher surface might be more retentive than the smoother surface for the non-adhesive acrylic resin, clinicians should note that mechanical properties of Ag-Pd-Cu-Au alloy are inferior to those of Ti-6Al-7Nb alloy (17), even if Ag-Pd-Cu-Au alloy has been age-hardened.

Statistical analyses also revealed that post-thermocycling bond strength of the TBB resin to the Ag-Pd-Cu-Au alloy was significantly higher than that to the Ti-6Al-7Nb alloy for all three surfaces (c>h, d>i, and e>j). Since the resin material did not contain any functional monomer in the composition, this could be explained by the difference in surface texture of the alloys. Among the three surface preparations, Ag-Pd-Cu-Au alloy appeared to be rougher than Ti-6Al-7Nb alloy when the surface was abraded with 0.6 MPa air-pressure (Figs. 2b and 3b). The difference in surface roughness between the two alloys is consistent with the difference in post-thermocycling bond strength reported

here.

Overall, shear bond testing results demonstrated the effectiveness of alumina air-abrasion for bonding to both the casting alloys. The question of the bonding durability of mechano-chemical retention systems remains, and needs to be evaluated. On the basis of the current study, the application of alumina air-abrasion with 0.6 MPa pressure is recommended for bonding the TBB-initiated acrylic resin to the two casting alloys.

Acknowledgments

This work was supported in part by Grant-in-Aid for Scientific Research C 20592302 (2008) from the Japan Society for the Promotion of Science (JSPS), Special Research Grant for the Development of Distinctive Education from the Promotion and Mutual Aid Corporation for Private School of Japan (2008), a grant from Dental Research Center A (2008), and Sato Fund (2008), Nihon University School of Dentistry.

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