

Effect of alumina abrasion for bonding between Ti-6Al-7Nb alloy and an indirect composite

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Purpose: The current study aimed to examine the influence of air-pressure and alumina grain size in air-abrasion on bonding between an indirect prosthodontic composite material and a titanium alloy.

Materials and Methods: Disk specimens were cast from a titanium-aluminum-niobium alloy (Ti-6Al-7Nb, T-Alloy Tough) and air-abraded with eight different air-pressures (0.00-0.40 MPa) and four different sizes of alumina particles (50-250 μm). The disks were bonded with a composite material (Estenia) and 24-hour shear bond strengths were determined.

Results: Among the eight air-pressure conditions employed for abrading 50 μm alumina, the greatest bond strengths (33.7-38.7 MPa) were recorded with air-pressures of 0.30, 0.35, and 0.40 MPa ($p < 0.05$). When the alloy was air-abraded with 0.35 MPa pressure using four different sized alumina, the group abraded with 50 μm alumina showed the highest bond strength (38.7 MPa, $p < 0.05$).

Conclusion: Within the study's limitations, the use of 50 μm particle sized alumina in combination with the air-pressure of 0.35 MPa is recommended for bonding the Estenia composite material to Ti-6Al-7Nb alloy. (Int Chin J Dent 2004; 4: 8-14.)

Clinical Significance: Air-pressure and alumina grain size in air-borne particle abrasion influence the bonding characteristics between an indirect composite and a titanium alloy.

Key Words: air-abrasion, alumina, bonding, niobium, Ti-6Al-7Nb alloy, titanium.

Introduction

The development of laboratory techniques, along with mould materials and casting machines, has led to titanium and titanium alloys being used for cast restorations and denture frameworks. Although the majority of titanium castings function successfully,¹⁻³ several problems worthy of attention have been reported. One of these is the insufficiency of the mechanical properties of titanium to fabricate long-spanned fixed partial dentures and removable denture frameworks. Titanium alloys have been developed as alternatives to titanium for improved mechanical properties in an oral environment. Among the titanium alloys reported for medical and dental use, a titanium-aluminum-niobium (Ti-6Al-7Nb) alloy seems to be the most suitable for dental castings because of its improved mechanical properties and corrosion resistance. The Ti-6Al-7Nb alloy, originally developed as an orthopaedic implant material,⁴ has been evaluated as a dental material. It was concluded that its mechanical properties were comparable to and corrosion resistance was superior to a titanium-aluminum-vanadium (Ti-6Al-4V) alloy.^{5,6} Additionally, the Ti-6Al-7Nb alloy was successfully cast in a magnesia-based mould material using a centrifugal casting apparatus.⁵

Bonding between polymer materials and titanium has been improved considerably by use of silane⁷ or

titanate⁸ couplers, and carboxylic or phosphoric acid derivatives,⁸⁻¹⁴ whereas surface modification techniques were reported to be effective for bonding titanium and the Ti-6Al-4V alloy.¹⁵⁻²¹ For the Ti-6Al-7Nb alloy, both a phosphate monomer²²⁻²⁴ and a surface modification technique²² have improved bonding between the alloy casting and polymeric materials.

Prior to the bonding procedure, the priming and surface modification systems require air-abrasion of alumina to mechanically clean the surfaces and to increase the surface bonding area. Therefore, with Ti-6Al-7Nb, it is necessary to set up an appropriate condition 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,²⁵⁻²⁹ no information is available about the Ti-6Al-7Nb alloy. The purpose of the current study was to evaluate the influence of different air-pressures and alumina grain sizes for air-borne particle abrasion on the resulting bond strength between a Ti-6Al-7Nb alloy and an indirect prosthodontic composite.

Materials and Methods

A titanium alloy containing 7.0% niobium and 6.0% aluminum (Ti-6Al-7Nb, T-Alloy Tough, GC Corp., Tokyo, Japan) designed for cast restorations and partial denture frameworks was selected for the substrate material. Four alumina materials with different particle sizes (50 μm , Hi-Aluminas, Shofu Inc., Kyoto, Japan; 90 μm , Aluminum Oxide, Danville Engineering, San Ramon, CA, USA; 125 μm , WA100, Kulzer Inc., Irvine, CA, USA; and 250 μm , W100, Kulzer Inc.) were used for air-abrasion. An indirect prosthodontic composite (Estenia, Kuraray Medical Inc., Tokyo, Japan) was employed as the veneering agent. Details of the materials are summarized in Table 1.

A total of 88 disk specimens, 10 mm in diameter and 2.5 mm thick, were prepared using a magnesia-based investment material (Selevest CB, Selec, Osaka, Japan) and a centrifugal casting apparatus (Ticast Super R, Selec). All disks were sanded with 600-grit silicon-carbide abrasive paper and divided into two groups.

Group 1. Air-abrasion with Different Air-pressures

Sixty-four disks were divided into eight sets of eight disks. Of these, seven groups were air-abraded with the 50- μm alumina by means of an air-borne particle abrader for 15 s (CL-FSG94, Kulzer, Inc.) with different air-pressures; 1) 0.10 MPa, 2) 0.15 MPa, 3) 0.20 MPa, 4) 0.25 MPa, 5) 0.30 MPa, 6) 0.35 MPa, and 7) 0.40 MPa. The distance of the orifice from the metal surface was approximately 20 mm. The remaining group was used as the control without air-abrasion. The Estenia Opaque Primer (Kuraray Medical Inc.) agent was applied to the metal surface with a sponge pallet. After surface preparation, a piece of double-coated tape with a circular hole 5 mm in diameter was positioned on each disk specimen to define the bond area. Two thin layers of an opaque resin (Estenia Opaque, OA3; Kuraray Medical Inc.) were applied to the primed area metal surface and each layer was separately light-exposed for 30 s in a laboratory light-polymerizing unit (Alpha light II; J. Morita Corp., Osaka, Japan). A brass ring (6 mm inside diameter, 2 mm height and 1 mm wall thickness) was placed so as to surround the opaque resin. The

ring was filled with the Estenia composite material (DA3), and light-polymerized with the unit for 30 s. The specimens were heated in an oven (KL-310, Kuraray Medical Inc.) at 100°C for 15 minutes. Eight specimens for each surface preparation group were prepared. Thirty minutes after preparation, the specimens were immersed in 37°C water for 24 hours before testing for bond strength.

Table 1. Details of casting alloy, alumina powder and indirect composite system assessed.

Material/Trade name	Manufacturer	Component
Casting alloy		
T-Alloy Tough	GC Corp., Tokyo, Japan	86.5 Ti, 7.0 Nb, 6.0 Al, mass%
Alumina Powder		
Hi-aluminas	Shofu, Kyoto, Japan	50 µm
Aluminum Oxide	Danville Engineering, San Ramon, CA, USA	90 µm
WA100	Kulzer Inc., Irvine, CA, USA	125 µm
W100	Kulzer Inc.	250 µm
Indirect composite system		
Estenia	Kuraray Medical Inc., Tokyo, Japan	Opaque OA3 Dentin DA3
Estenia Opaque Primer	Kuraray Medical Inc.	MDP and initiator in solvent

MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

Group 2. Air-abrasion with Different Particle-sized Alumina

Twenty-four disks were divided into three sets of eight. Each set was air-abraded with one of the three sizes of alumina particles (90, 125, and 250 µm) using the CL-FSG94 abrader for 15 s at 0.35 MPa air-pressure, and then fabricated as described for Group 1.

Each specimen was embedded in an acrylic resin mould and seated in an ISO 11405 shear-testing jig. Shear bond strengths were then determined with a mechanical testing machine (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 0.5 mm/minute. The average bond strength and the standard deviation (SD) of eight replications were calculated for each condition. The results were analyzed by analysis of variances (ANOVAs) and the post-hoc Duncan new multiple range test. The value of statistical significance was set at 0.05.

The Ti-6Al-7Nb disk surfaces air-abraded with the four different particle sized alumina were observed with a scanning electron microscope (SEM; S-3500N, Hitachi Corp., Tokyo, Japan) operated at 20 kV.

Results

Table 2 presents the ANOVA results. One-way ANOVAs run on the shear testing results showed statistical significance for both air-pressure (Group 1, $F=9.595$, $p=0.0001$) and difference in particle size (Group 2, $F=13.493$, $p=0.0001$). The bond strength results were therefore analyzed using a post-hoc Duncan new multiple range test calculated at the 0.05 significance level.

Table 3 and Figs. 1-2 indicate the results of shear testing and statistically categorized groupings. Bond

strength of Group 1 air-abraded with 50 μm alumina varied from 17.6 to 38.7 MPa according to the different air-pressures. Duncan new multiple rang test divided the results of the Group 1 into five groupings; (a)-(e). The 0.30 MPa (grouping d, e), 0.35 MPa (grouping e), and 0.40 MPa (grouping d, e) groups were the greatest, followed by the 0.25 MPa (grouping c, d), 0.20 MPa (grouping b, c), and 0.15 MPa (grouping b, c, d) groups. The 0.10 MPa and no-abrasion groups resulted in the lowest bond strengths (grouping a).

Table 2. Analysis of variance (ANOVA) results.

Source	Degree of freedom	Sum of squares	Mean square	F-value	P-value
One-way ANOVA for the different air-pressure group					
Air pressure	7	2643.223	377.603	9.595	0.0001
Residual	56	2203.726	39.352		
One-way ANOVA for the different particle size group					
Particle size	3	701.681	233.894	13.493	0.0001
Residual	28	485.350	17.334		

Table 3. Shear bond strength results and statistically categorized groupings.

Air-pressure variation (Alumina particle size: 50 μm)				Alumina particle size variation (Air-pressure: 0.35 MPa)			
	Mean (MPa)	SD	Grouping *		Mean (MPa)	SD	Grouping*
None	17.6	3.8	a	50 μm	38.7	6.0	B
0.10 MPa	22.4	5.0	a	90 μm	30.8	2.7	A
0.15 MPa	28.5	6.5	b, c, d	125 μm	27.9	4.4	A
0.20 MPa	26.0	4.7	b, c	250 μm	26.7	2.7	A
0.25 MPa	30.9	5.9	c, d				
0.30 MPa	33.7	8.6	d, e				
0.35 MPa	38.7	6.0	e				
0.40 MPa	34.2	8.5	d, e				

SD, Standard deviation; Grouping, Identical letters indicate that the values are not statistically different ($p > 0.05$).

Bond strength of Group 2 abraded with 0.35 MPa air-pressure varied from 26.7 to 38.7 MPa according to the different alumina particle sizes. Shear testing results of Group 2 showed that the greatest bond strength was achieved through the use of 50 μm grain sized alumina (grouping B). The other three groups (90, 125, and 250 μm) showed statistically lower bond strength than the 50 μm group; the differences among these three groups were not statistically significant (grouping A).

Fig. 3 shows Ti-6Al-7Nb alloy surfaces after air-abrasion with four different particle sized alumina; the surfaces of the specimens were observed to become rougher in accordance with their particle size.

Discussion

The current study compared the effects of different air-pressures and alumina grain sizes for air-borne particle abrasion on bonding between a Ti-6Al-7Nb alloy and an indirect prosthodontic composite material. Before application of a phosphate monomer contained metal conditioning agent, the substrate alloy surfaces

were air-abraded with alumina to mechanically clean the surfaces and to increase the surface bonding area. We did not use additional mechanical retention systems to evaluate the effect of air-abrasion of the air-pressure and particle sized of alumina.

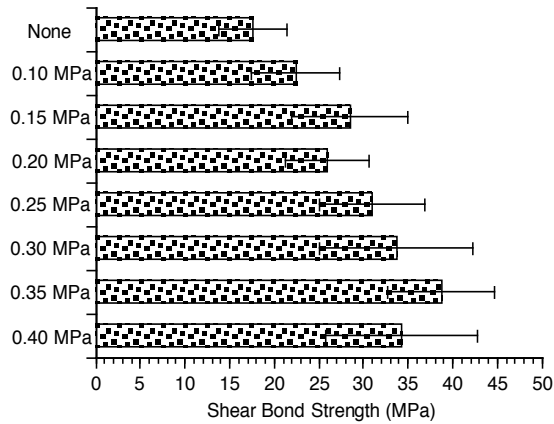


Fig. 1. Shear testing results for different air-pressure (50 μ m grain sized alumina).

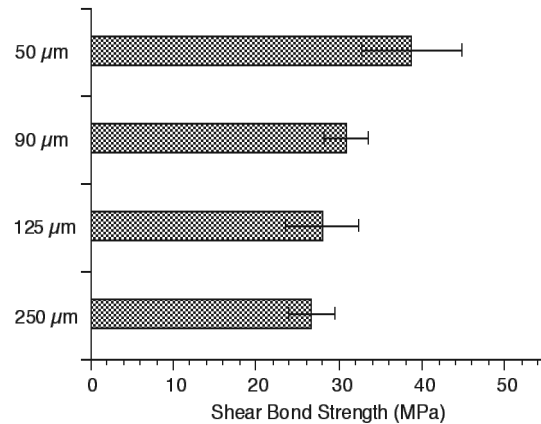
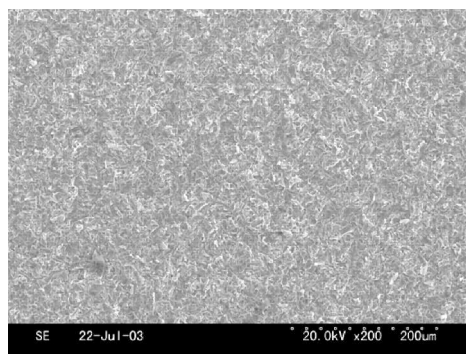
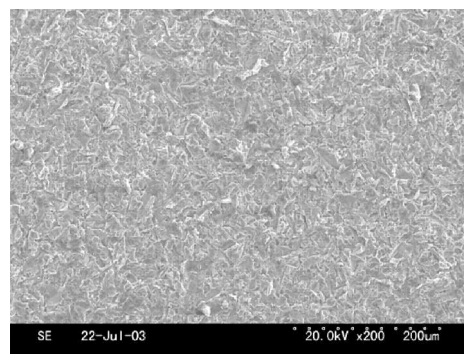


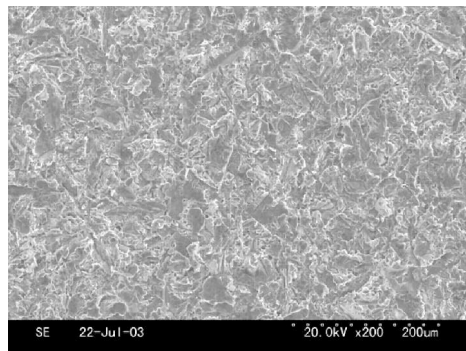
Fig. 2. Shear testing results for different grain sized alumina (0.35 MPa air-pressure).



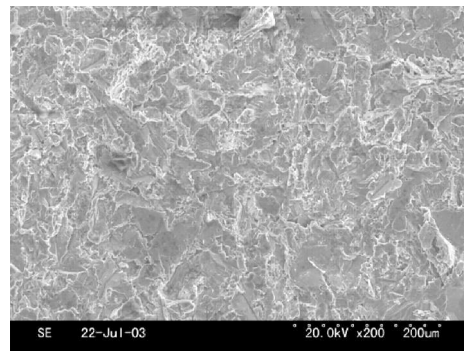
50 μ m



90 μ m



125 μ m



250 μ m

Fig. 3. Scanning electron micrographs of Ti-6Al-7Nb alloy after air-abrasion with alumina.

Shear testing of Group 1 air-abraded with 50 μ m alumina revealed that the 0.30, 0.35 and 0.40 MPa exhibited considerably greater bond strengths than the other air-pressures. The results reveal the necessity of air-abrasion before bonding procedures, and that the 0.30, 0.35 and 0.40 MPa air-pressures were suitable for the Ti-6Al-7Nb alloy. In the Group 1 study, the air-pressure of 0.35 MPa recorded the highest bond strength, and statistical grouping divided it into the statistically highest group (group e).

In the group 2 study results, air-abrasion with different particle sized alumina, show that the 50 μm sized alumina recorded greater bond strength than the other three particle sized alumina. Another study has reported that the types of alloy and the particle size used for air-abrading influenced wettability.²⁵ The wettability of an alloy surface is an important factor for bonding between polymeric materials and metal; better wetting with metal surface conditioner and resin materials affects the interlocking between the resin and the metal surface. The importance of the wettability can be seen in the reported low bond strength of the direct relining material with the spatulation technique; the authors speculating that this was due to the high viscosity of the material in the resin-metal bonding procedure, as well as to insufficient interlocking between the resin and the roughened metal surface.²³ On the particle sizes of alumina, another study for highly pure titanium reported that air-abrading with 250 μm sized alumina was effective for bonding;²⁸ another report described that the use of a large particle size alumina seems to be advantageous in reducing the weight of alumina remaining on the titanium surface, while increasing surface roughness.²⁷ According to SEM observations in the current study, the larger particle sized alumina also caused a rougher surface, and a surface air-abraded with the 50 μm particle showed a mossy appearance (Fig. 3). Nevertheless, the results of wear resistance of the Ti-6Al-7Nb alloy evaluated here showed it to be significantly higher than highly pure titanium grades 2 and 3.³⁰ The large size alumina might not therefore affect bonding of the Ti-6Al-7Nb alloy. It was hypothesized that surfaces roughened with the 50 μm sized alumina were in suitable condition of wettability for a phosphate conditioner and a composite opaque resin, and recorded the highest bond strengths.

Our shear testing results clearly demonstrated the effectiveness of air-abrasion for bonding titanium alloy. A question concerning the bonding durability remains, and needs to be evaluated. On the basis of the current study, we recommend the use of the combination of 0.35 MPa air-pressure with a 50 μm particle sized alumina for air-abrading before bonding an indirect prosthetic composite material and a Ti-6Al-7Nb alloy framework.

Conclusion

Within the limitations of the current experimental settings, the following conclusions can be drawn:

1. The 24-hour bond strength between a Ti-6Al-7Nb alloy (T-Alloy Tough) and an indirect composite material (Estenia) was improved by application of 50 μm alumina air-abrasion with air-pressure ranging from 0.15 to 0.40 MPa.
2. Statistically, the greatest bond strength was recorded using 0.30 to 0.40 MPa air-pressure conditions in combination with the 50 μm alumina particles.
3. When an identical air-pressure (0.35 MPa) was employed, the use of 50 μm alumina exhibited the greatest bond strength.

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