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RESEARCH ARTICLE

EFFECT OF SURFACE TREATMENT ON INTERFACIAL SHEAR BOND STRENGTH OF LOW FUSING CERAMIC TO COMMERCIALLY PURE TITANIUM

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ABSTRACT

Objectives: To evaluate interfacial shear bond strength between commercially pure titanium and low fusing ceramic after surface treatments of titanium.
To determine which surface treatment was superior and can provide interfacial shear bond strength value comparable with nickel chromium and low fusing ceramic.
Methods: 88 discs of titanium and 10 discs of nickel chromium were obtained and the titanium discs were subjected to the following surface treatments: no surface treatment, sand blasted with alumina particles (250µm), acid etched in a HNO_3/HF solution, bonding agent, sandblasting and acid treatment, acid etching and bonding agent, sandblasting plus bonding agent application, all surface treatments.
After treatments, discs were ultrasonically cleaned and ceramic was fired. They were embedded in acrylic blocks and universal testing machine was used to obtain bond strength values. One disc from each group was sent for study of surface topography using scanning electron microscope.
Results: The bond strength values of all groups were statistically significant. The mean interfacial shear bond strength was highest in nickel chromium group followed by the group that was sand blasted and bonding agent. The least bond strength value was seen in the group that received no treatment.
Significance: Sandblasting and use of bonding agent result in enhanced bonding between commercially pure titanium and low fusing ceramic not comparable to the bond strength provided by nickel chromium. Further studies have to be done for a more suitable surface treatment that will result in a bond strength comparable to that between nickel chromium and ceramic.

INTRODUCTION

The use of porcelain fused to metal fixed dental prosthesis is still considered a viable option for oral rehabilitation owing to its mechanical strength. The obvious advantages of mechanical properties of non precious metal alloys permits for fabrication of restorations with higher rigidity and lesser thickness.[1] Pure titanium has had a role to play in porcelain fused to metal fixed dental prostheses in the last decade. When weighed against cast metal alloys, pure titanium has superior biocompatibility, more desirable mechanical properties, higher strength, greater availability and an affordable price. [2] Titanium by nature is highly reactive element. Oxygen reacts spontaneously with titanium at room temperature, due to its high affinity, forming an oxidized surface. [3]

The metal- ceramic bond interface is critical for the clinical success of porcelain fused to metal restorations. The strength of the porcelain to metal bond determined by the oxide layer formed on the surface of the metal, mechanical interlocking, Van der Waals forces and compressive forces originating from the coefficient of thermal expansion. Among these factors, coefficient of thermal expansion can create strong shear stresses at the porcelain metal interface. The clinical life of porcelain fused to metal restorations depends on the formation of this layer. [1] In a porcelain metal system, the thermal expansion coefficients must be matched to ensure optimum bond strength. In order to reduce titanium oxidation at high temperatures, low fusing porcelains were introduced that are capable of bonding to titanium at temperatures below 850° Celsius. [2]

Surface treatments of metal such as acid treatments, airborne particle abrasion etc form a roughened metal surface thereby

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enhancing the surface area contacting the porcelain and thus bringing about an increase in the oxide per unit area. This in turn improves upon the bond between metal and ceramic on a chemical basis, not to mention the increased wettability of porcelain to metal, providing better mechanical interlocking of porcelain to metal. [2]

MATERIALS AND METHODS

Commercially pure titanium rods were sectioned into 88 discs measuring 10mm in diameter and 5mm in height (Rajasthan Industries, Bangalore). Prior to receiving the surface treatment, the discs were polished with #1200 grit silicon carbide paper. 8 discs were subjected to surface treatment and SEM imaging whereas the remaining 80 specimens were divided into eight groups of 10 each to receive only surface treatments. (Figure 1) After each surface treatment, all the specimens were cleaned ultrasonically in an ultrasonic cleaner (CD4280, Codyson Digital Ultrasonic Cleaner) with distilled water for ten minutes. Excess water was removed and the metal specimens were dried at room temperature for thirty minutes.

The control **Group A** was polished with #1200 grit silicon carbide paper. A fresh piece of silicon carbide paper was used for each disc. Polishing was done at a speed of 600 rpm. (Figure 2). **Group B** was sandblasted with alumina particles (Strahlmittel, Renfert) of size 250µm for twenty seconds at a pressure of 4 bar and held at an angle of 45° to the nozzle of the sand blasting machine.(Figure 3). **Group C** specimens were etched with17%HNO_3/HF solution (Emplura, Merck Chemicals) for five minutes and the surfaces were cleaned with distilled water and pat dried. (Figure 4). In **group D** a layer of bonding agent (Ti Bond, GC) was applied on the metal surface of uniform thickness and fired in the ceramic furnace (Programat P300, IvoclarVivadent). (Figure 5) **Group E** specimens were subjected to sandblasting followed by acid etching.

Specimens belonging to **Group F** received a combination of acid etching and a layer of bonding agent. **Group G** was sandblasted followed by application of bonding agent. **Group H** specimens received all three surface treatments in the order of sand blasting, acid etching and finally bonding agent. **Group I** included ten nickel chromium specimens that were subjected to sand blasting with alumina particles (Strahlmittel, Renfert) of size 250µm for twenty seconds at a pressure of 4 bar and held at an angle of 45° to the nozzle of the sand blasting machine.

After the surface treatments were completed successfully, low fusing ceramic (Initial, GC) was applied. All specimens were preheated following which they received a layer of dentin opaque, dentin and finally enamel. Each of the layers was fired individually before application of the next layer.

The final height of the ceramic achieved was 5mm with an 8mm diameter. The firing programme for each layer is as follows:

Acrylic blocks were fabricated after ceramic firing onto which the discs could be individually embedded. A wax block of dimensions 3cm x 3cm x 5cm was fabricated with modelling wax (Hindustan Dental Products). Putty consistency elastomeric impression material (Elite P & P, Zhermack) was adapted around this wax block evenly to a thickness of 1cm all around it. Once the impression material set, the wax block was removed and orthodontic wire (Leone, Germany) of 1mm gauge and 6cm length was centred on the ceramic of the disc and stabilised with sticky wax (Dental Products of India). This was then positioned over the putty mold and stabilised. Auto polymerising acrylic was then mixed according to manufacturer's instructions and poured into the mold space till it embedded the disc within it. The acrylic block so obtained was removed from the mold and irregularities were trimmed. (Figure 6) All 80 acrylic blocks were fabricated in a similar manner. Each of the specimens was fastened by the jig of the universal testing machine (Multitest 10-i, Mecmesin) and a blade was used with a cross head speed of 1mm/minute at the junction of the ceramic and the metal disc. Load was applied till separation of the ceramic from the disc was achieved. The value at this point was noted in mega pascals. The remaining 8 titanium discs which received only surface treatment were used for obtaining SEM images (Gemini, Ziess). Each of the specimens was loaded onto a mounting plate and gold sputtering was done. Following this, the specimens were placed into the scanning electron microscope and images of the surface were obtained at 5kx, 10kx and 20kx magnification. (Figure 7)

RESULTS

The specimens were subjected to various surface treatments and then secured onto the acrylic blocks according to the specifications suggested by the Universal Testing Machine operator. The acrylic blocks were subjected to interfacial shear bond test in the universal testing machine (Multitest 10 i, Mecmesin). The remaining eight titanium discs were observed for surface roughness with a scanning electron microscope.

The data collected was entered into a Microsoft excel spreadsheet and analysed using IBM SPSS Statistics, Version 22(Armonk, NY: IBM Corp). The comparison of the mean interfacial shear bond strength of each of the groups is depicted in table 1. The values ranged from a minimum value of 8.28Mpa to 16.09Mpa. Group C had the least mean interfacial shear bond strength value while Group I had the highest value. The p value obtained was less than 0.001.

	Preheating Temperature	Drying Time	Raise of Temperature	Vacuum	Final Temperature	Holding Time
Ti Bonder firing	450°C	4 Minutes	55°C/min	Yes	810°C	1minute
Opaque firing	450°C	4 Minutes	55°C/min	Yes	810°C	1minute
Dentin and Enamel Firing	450°C	4 Minutes	55°C/min	Yes	810°C	1minute

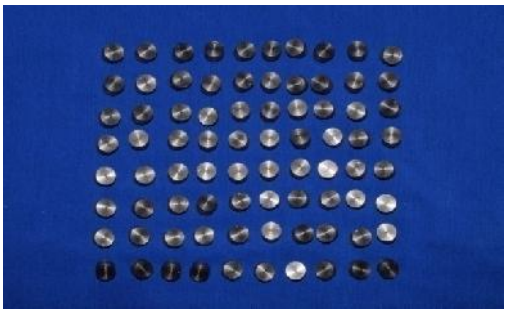


Fig. 1 Titanium discs obtained after sectioning

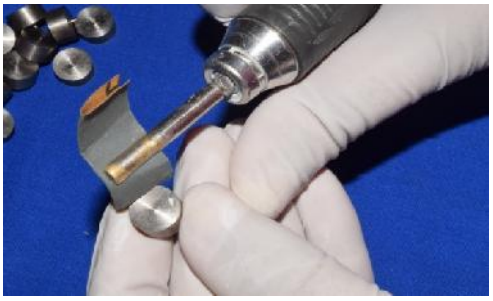


Fig. 2 Titanium discs polished with silicon carbide paper

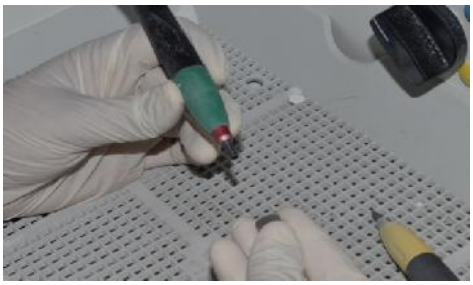


Fig. 3 Sandblasting titanium discs

Fig. 4: 17%HNO_3/HF solution

Fig. 5 Low fusing ceramic system with Bonding agent

Fig. 6 Finished acrylic blocks

This indicated that the mean interfacial shear bond strength values were statistically different among the groups ($p<0.05$). Table 2 interprets the comparison between the mean interfacial shear bond strength of the specimens in Group A with the specimens of the remaining other groups. The Bonferroni post hoc test revealed the following: the mean interfacial shear bond strength of specimens that received no surface treatment was less than Group B by 3.434Mpa. The mean interfacial shear bond strength of Group D was superior to the group that received no surface treatment. Group A was less effective than the Group D by 6.33Mpa.

Table 1 Comparison of Mean Interfacial Shear Bond Strength Among All Groups

GROUP	N	Mean	Std. Deviation	ANOVA	
				F(df1,df2)	p-value
No treatment	10	8.55	1.16		
Sand blasting (SB)	10	11.98	1.87		
Acid etching (AE)	10	8.28	1.63		
Bonding agent (BA)	10	14.88	2.05		
SB+ AE	10	13.04	1.17	39.69(8,81)	<0.001*
AE + BA	10	9.70	1.50		
SB + BA	10	15.24	1.29		
SB+AE+BA	10	8.52	1.60		
Nickel-chromium group	10	16.09	1.69		

*p<0.05 Statistically significant
p>0.05 Non significant, NS

Table 2 Comparison Between Mean Interfacial Shear Bond Strength Of Group A With Remaining Other Groups

Group	Group	Mean Difference	Std. Error	95% Confidence Interval		p-value
				Lower Bound	Upper Bound	
No treatment	Sand blasting (SB)	-3.434	0.70	-5.77	-1.09	<0.001*
	Acid etching (AE)	0.26	0.70	-2.07	2.60	1.00(NS)
	Bonding agent (BA)	-6.33	0.70	-8.68	-3.99	<0.001*
	SB+ AE	-4.49	0.70	-6.83	-2.14	<0.001*
	AE + BA	-1.15	0.70	-3.49	1.19	1.00(NS)
	SB + BA	-6.69	0.70	-9.03	-4.34	<0.001*
	SB+AE+BA	0.03	0.70	-2.31	2.37	1.00(NS)
	Nickel-chromium	-7.54	0.70	-9.88	-5.20	<0.001*

Bonferroni Post hoc test
*p<0.05 Statistically significant, p>0.05 Non significant, NS

The mean interfacial shear bond strength values of the Group A and Group C was found to be statistically insignificant. In the case of the combined surface treatment groups, statistically insignificant values were obtained when compared with Group F and with the Group H. The Group G and Group E were

superior to the Group A by 6.69Mpa and 4.49Mpa respectively. Group I was superior to the Group A by 7.54Mpa.

The comparison between the mean interfacial shear bond strength among Group I and each of the remaining other groups is represented in table 3.

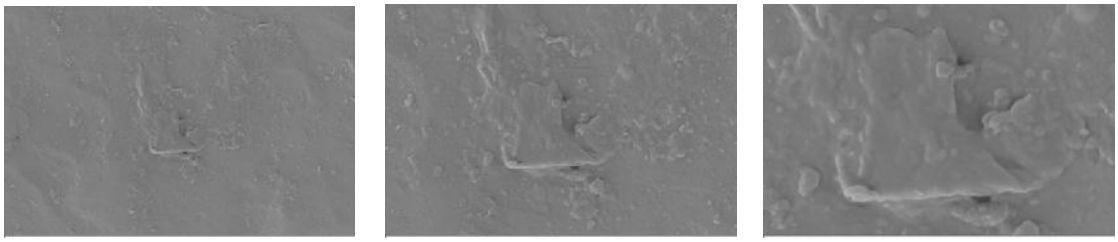


Fig. 7 (a): SEM images of Group A

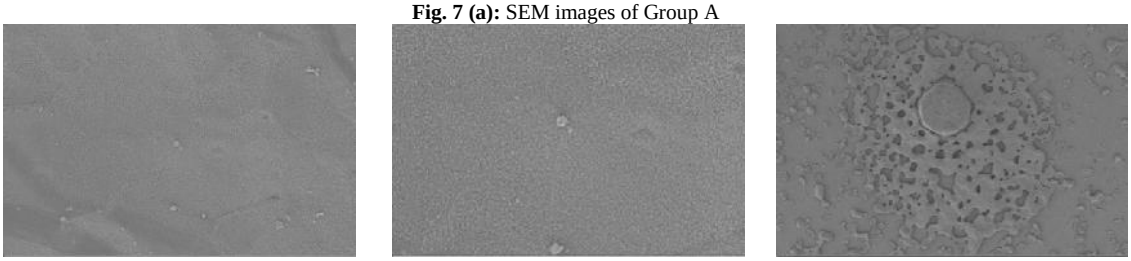


Fig. 7(b): SEM images of Group B

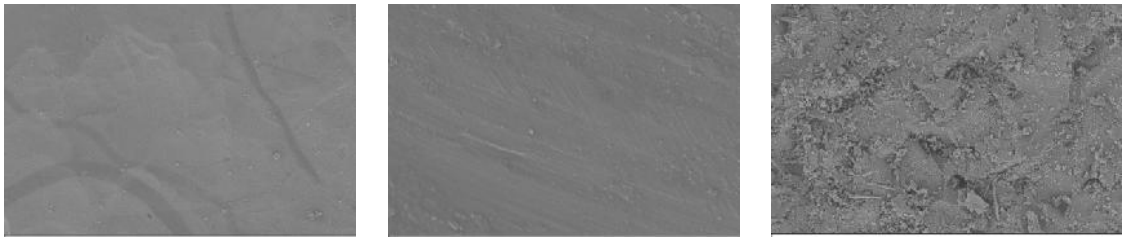


Fig 7(c): SEM images of Group C

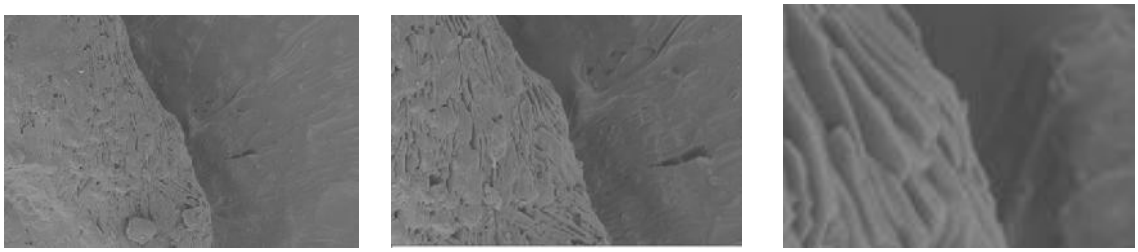


Fig 7(d): SEM images of Group D

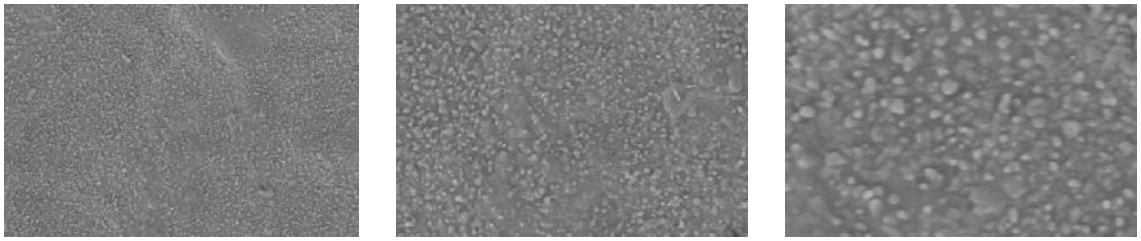


Fig 7(e): SEM images of Group E

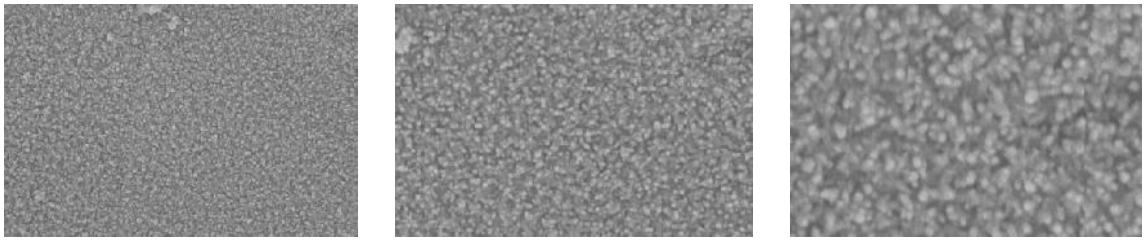


Fig 7(f): SEM images of Group F

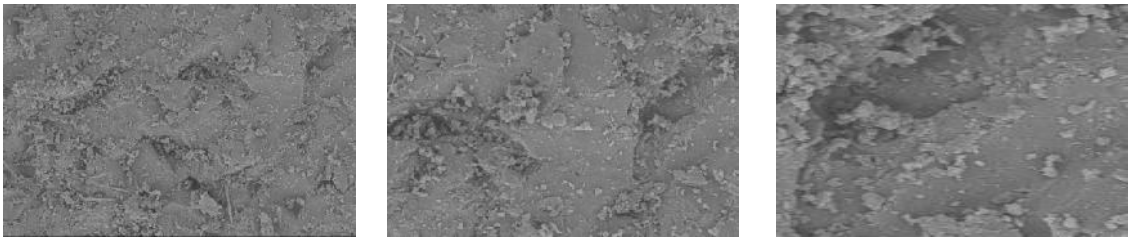


Fig 7(g): SEM images of Group G

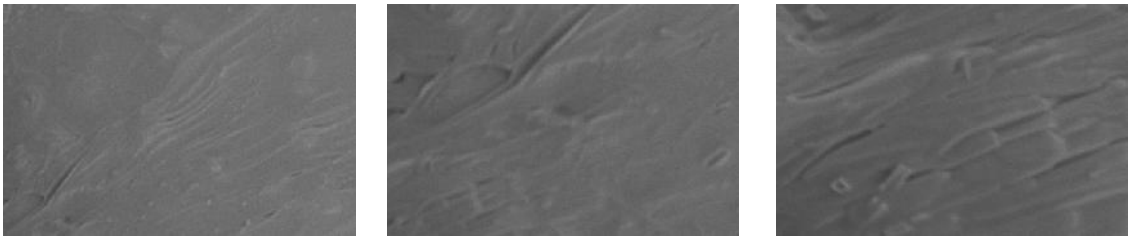


Fig 7(h): SEM images of Group H

Table 3 Comparison between Mean Interfacial Shear Bond Strength Of Group I With Remaining Other Groups

Group	Group	Mean Difference (Mpa)	Std. Error	95% Confidence Interval		p-value
				Lower Bound	Upper Bound	
Nickel-chromium group	Sand blasting (SB)	4.11	0.70	1.76	6.45	<0.001*
	Acid etching (AE)	7.80	0.70	5.46	10.15	<0.001*
	Bonding agent (BA)	1.20	0.70	-1.13	3.54	1.00(NS)
	SB+ AE	3.05	0.70	0.71	5.39	0.002*
	AE + BA	6.39	0.70	4.05	8.73	<0.001*
	SB + BA	0.85	0.70	-1.48	3.19	1.00(NS)
	SB+AE+BA	7.57	0.70	5.23	9.91	<0.001*

Bonferroni Post hoc test
*p<0.05 Statistically significant , p>0.05 Non significant, NS

It was found that Group I was generally superior in mean interfacial shear bond strength to the remaining other groups. It was superior to the Group B and Group C by 4.11Mpa and 7.80Mpa respectively. The group D had a mean interfacial shear bond strength value inferior to Group I by 1.20Mpa which was statistically insignificant. Among the groups that received combination treatments, Group I was found to be superior to Group E, Group F and Group G by 3.05Mpa, 6.39Mpa and 0.85Mpa respectively. Group I was also found to be superior to Group H by 7.57Mpa. The bond strength between Group I and Group G were found to be comparable. Table 4 interprets the comparison between the mean interfacial shear bond strength of each surface treated titanium group with the remaining other groups. Group B was compared with the other groups.

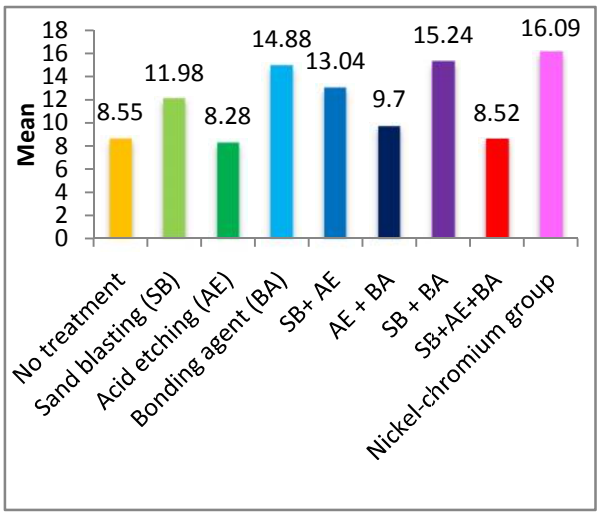
Table 4 Comparison Between Mean Interfacial Shear Bond Strength Of Each Surface Treated Titanium Group With Remaining Surface Treated Titanium Groups

Group	Group	Mean Difference (Mpa)	Std. Error	95% Confidence Interval		p-value
				Lower Bound	Upper Bound	
Sand blasting (SB)	Acid etching (AE)	3.69	0.70	1.35	6.04	<0.001*
	Bonding agent (BA)	-2.90	0.70	-5.24	-0.56	0.003*
	SB+ AE	-1.05	0.70	-3.39	1.28	1.00(NS)
	AE + BA	2.28	0.70	-0.05	4.62	0.065
	SB + BA	-3.25	0.70	-5.60	-0.91	0.001*
	SB+AE+BA	3.46	0.70	1.12	5.80	<0.001*
Acid etching (AE)	Bonding agent (BA)	-6.60	0.70	-8.94	-4.25	<0.001*
	SB+ AE	-4.75	0.70	-7.09	-2.41	<0.001*
	AE + BA	-1.41	0.70	-3.75	0.92	1.00(NS)
	SB + BA	-6.95	0.70	-9.29	-4.61	<0.001*
	SB+AE+BA	-.23	0.70	-2.57	2.10	1.00(NS)
Bonding agent (BA)	SB+ AE	1.84	0.70	-0.49	4.19	0.38(NS)
	AE + BA	5.18	0.70	2.84	7.53	<0.001*
	SB + BA	-.35	0.70	-2.69	1.98	1.00(NS)
	SB+AE+BA	6.36	0.70	4.02	8.71	<0.001*
SB+ AE	AE + BA	3.34	0.70	0.99	5.68	<0.001*
	SB + BA	-2.20	0.70	-4.54	0.14	0.09(NS)
	SB+AE+BA	4.52	0.70	2.17	6.86	<0.001*
AE + BA	SB + BA	-5.54	0.70	-7.88	-3.19	<0.001*
	SB+AE+BA	1.18	0.70	-1.16	3.52	1.00(NS)
SB + BA	SB+AE+BA	6.72	0.70	4.37	9.06	<0.001*

Bonferroni Post hoc test
*p<0.05 Statistically significant, p>0.05 Non significant, NS

It was found that Group B was superior to Group C, Group F and Group H by 3.69Mpa, 2.28Mpa and 3.46Mpa respectively. It was inferior to Group D and Group G by 2.90Mpa and 3.25Mpa respectively. Group B had a mean interfacial shear bond strength that was comparable to the Group E (1.05Mpa).

When Group C was compared with the other groups, the following results could be inferred.



Graph 1 Mean Interfacial Shear Bond Strength Among Each Of The Groups

This group was found to be generally inferior in mean interfacial shear bond strength to Group D, E, F, G as well as the Group H by 6.60Mpa, 4.75Mpa, 1.41Mpa, 6.95Mpa and 0.23Mpa respectively. The mean interfacial shear bond strength of the Group C was found to be comparable with that of Group F and the Group H. Comparison of Group D with the other combination groups revealed the following: the mean interfacial shear bond strength of this group was found to be superior to Groups E, F and H by 1.84Mpa, 5.18Mpa and 6.36Mpa respectively. Though it was found to be inferior to Group G by 0.35Mpa, this was found to be statistically insignificant. Group E was compared with the other three combination groups. It was found that the mean interfacial shear bond strength of this group was superior to Group F and the Group H by 3.34Mpa and 4.52 Mpa respectively. This group was found to have a mean interfacial shear bond strength value inferior to that of the Group G by 2.20Mpa which was statistically insignificant. Group F was compared with the other combination groups.

It was found to be inferior to Group G by 5.54Mpa but comparable to the Group H (1.18Mpa). Group G was found to be superior to the Group H by 6.72Mpa.

The graph interprets the mean interfacial shear bond strength of each of the groups. The values ranged from a minimum value of 8.28Mpa to 16.09Mpa. The acid etched group had the least mean interfacial shear bond strength value while the nickel chromium group had the highest value of mean interfacial shear bond strength.

DISCUSSION

The study was conducted with the purpose of obtaining interfacial shear bond strength values between commercially pure titanium and low fusing ceramic after surface treatment of the titanium surfaces, and to compare these values with those obtained from nickel chromium fired with low fusing ceramic. The observational values were obtained using a universal testing machine (Multitest 10 i, Mecmesin), and the surface topography was studied using a scanning electron microscope (Gemini, Zeiss). All the values were recorded by a single operator in order to minimise the bias.

The bond between metal and ceramic may be evaluated with the help of various modes of testing such as three point bending, four point bending, biaxial flexural test and shear bond strength.[5,6,7,8,9] Ceramics are known for a higher resistance to compressive and tensile forces. However, a lower resistance has been reported toward shear forces.[10,11] Various literature support the theory that a roughened titanium surface can increase the bond strength between titanium and ceramic.[12,13] In this study, it was found that there was a statistically significant difference between the mean interfacial shear bond strength in each group. Group I showed the highest interfacial shear bond strength (16.09Mpa). Among the titanium groups, Group G showed the highest interfacial shear bond strength (15.24Mpa). The bond strength between metal and ceramic depends on chemical bond from metal oxide and mechanical bond with the surface irregularities on the metal surface. [14]

Ceramic firing is preferably performed at a temperature below 883°C. This is in order to bring about a reduction in the formation of the oxide layer on the surface of the titanium. [15] This study involves ceramic firing at temperatures that were below 810°C. Airborne particle abrasion achieves an increase in surface area. This helps improve the micro retentive property of the metal surface topography.[16] This in turn enhances the mechanical bond between titanium and ceramic.[17] In this study, aluminium oxide particle of diameter 250 µm (Strahlmittel, Renfert) was used to avoid embedding of these particles on the metal surface. A significant difference was noted between the mean interfacial shear bond strength of Group B when compared to the Group A. SEM imaging of the sand blasted titanium disc show a rough, irregular and acute angled titanium surface. According to Reyes *et al* (2001), acid etching the surface of metal produced a more ideal surface topography than sand blasting [15]. This is however in contrast with the present study which showed that acid etching was not as effective in improving bond strength as sand blasting. These results are in accordance with Sced and McLean (1973), who said that acid treatment hindered the layer of oxide that formed on the metal surface which may have affected the bonding between metal and ceramic. [16] SEM imaging of the acid etched specimen showed a smoother surface topography after acid treatment.

Application of bonding agent before sintering of ceramic on to titanium surfaces has significantly improved the bond between titanium and ceramic according to this study. Use of bonding

agent inhibits the formation of a non adherent oxide layer. This layer tends to form if the titanium is pre oxidated at high temperatures. Titanium particles get dispersed within the bonding agent. Here they behave as oxygen scavengers and enable prevention of a non adherent oxide layer from forming on the surface of the metal. [6,9] In this study, bonding agent used was compatible with the ceramic system. According to Derand and Hero (1992), a significant decrease in the bond strength was seen on application of bonding agent. This could be explained by the fact that the bonding agent and ceramic used in the study were of different manufacturers and therefore, may not have been compatible.[18] SEM image analysis of the specimen with a layer of bonding agent applied revealed a greater amount of surface roughness in its topography. In the current study, a significant enhancement was seen in the bond between metal and ceramic after acid treatment was done following sand blasting of the titanium specimens. The bond strength was significantly higher compared to the bond strengths of the individual treatment groups of sand blasting and acid etching. Although other surface treatments have been accounted for in various studies, they may be complex and expensive procedures [12]. In this study, simple and practical combination of surface treatments has proven to improve the bond.

Acid treatment followed by application of bonding agent failed to provide a significant enhancement in the interfacial shear bond strength between titanium and ceramic compared to the group that received no surface treatment. This is in agreement with a Hussaini and Wazzan (2005), who reported no improvement in bonding strength between titanium and ceramic. This indicates that the surface topography obtained after sand blasting is more preferred by the bonding agent when compared to the roughness resulting from acid treatment.[4] Sand blasting followed by use of bonding agent resulted in a surface that gave a significantly higher bond strength than all the remaining groups. The reason for this might be an enhanced wettablitity of the titanium surface upon treatment with bonding agent over the already sandblasted roughened surface. A study done by Hussaini and Wazzan also produced similar findings.

This study also attempted to combine sand blasting, acid etching and use of a bonding agent consecutively on the titanium surface to test for additive superiority over individual surface treatments. It was found that the resulting surface topography yielded a bond strength value that was barely comparable to the Group A. Possible reasons for this could be that the acid treatment prevented oxide layer formation on the titanium surface. Another plausible cause could be that the acid particles interfered with the action of the bonding agent with the surface particles of the titanium.

Limitations of this study are as follows: the use of a single brand of ceramic and the use of commercially pure titanium instead of titanium alloy prevents relatability of the findings of this study to other materials. Studying the mode of failure may also have given a better insight into the clinical applicability of this study.

CONCLUSION

Within the limitations of this study, the following conclusions may be drawn:

1. Surface treatment of titanium by sand blasting followed by application of bonding agent produced the highest interfacial shear bond strength values.
2. Surface treatment of titanium with acid, as an individual treatment or in combination with sand blasting and bonding agent application doe not significantly enhance the bond between ceramic and metal.
3. None of the surface treatments employed in this study were able to produce a shear bond strength that was comparable to the bond between nickel chromium and ceramic.

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