IOSR Journal of Dental and Medical Sciences (IOSR-JDMS)
e-ISSN: 2279-0853, p-ISSN: 2279-0861.Volume 19, Issue 5 Ser.2 (May. 2020), PP 17-23
www.iosrjournals.org

CAD/CAM restorations and its effect on bonding composite to Ti-6Al-4V alloy.

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Abstract
Context:*An appropriate method for veneering titanium abutment with esthetic material that has sufficient bond strength has not been determined. Various esthetic materials are frequently used to coat the surface of titanium abutment. A pilot study was conducted to assay the effects of provisional surface treatment using etchant and primer between a resin composite and Ti–6Al–4V alloy on bond strength*
Methods:*Discs of Ti–6Al–4V alloy was milled of 6mm diameter and 4 mm in height. The specimen was prepared by air abrasion with alumina and subsequently etching the alloy surface with 5wt% distilled water with ammonium hydrogen fluoride for 30 s. Former and latter to the etching process titanium alloy surface were examined under a lepton microscope. A 10-Methacryloxydcyl dihydrogen phosphate primer in succession with resin composite was put to the bonding area and veneered to the specimen respectively. The resin composite was further divided as either with or without milled-fiber resin composite. The specimen is treated at thermocycling 0 and thermocycling20,000. After thermocycling for 20,000 cycles, the specimen was analyzed utilizing ANOVA and a multiple comparison test ($\alpha = 0.05$). for bond strength.*
Results: *Highest bond strength of 27.2 MPa was shown when resin composite without fiber milling was used with f-etch and 10-Methacryloxydcyl dihydrogen phosphate primer, while resin composite without fiber milling and no etching with 10-Methacryloxydcyl dihydrogen phosphate primer showed to be the second regarding the bond strength of 25.2 MPa and followed by fiber milled resin composite, F-etch and 10-Methacryloxydcyl dihydrogen phosphate primer with a bond strength of 18.9 MPa, lesser bond strength of 16.8 MPa was shown by Fiber milled Resin Composite, no etching, and10-Methacryloxydecyl dihydrogen phosphate primer followed by 14.3 MPa with resin composite without fiber milling, no etching and no 10-methacryloxydcyl dihydrogen phosphate primer. However, the least value of 2.7MPa was shown with Fiber milled resin composite without acid etching and primer.*
Conclusion: *Ti-6Al-4V alloy and resin compositerecorded bond strength at its peak when the surface of the alloy was reformed with aluminum blasting, fluoride etchant, and phosphate primer while lowest bond strength was noted when aluminum blasting without any etchant or primer was used to adjust the alloy surface.*
Keywords: *Titanium alloy****,*** *fluoride etchant****,****phosphate primer,CAD/CAM systems*

Date of Submission: 25-04-2020 Date of Acceptance: 08-05-2020

I. Introduction

With the progress of a software and (CAD/CAM) systems, the utilization of metal in prosthetic restorations has inflated the use of titanium. Moreover, thecommercial pure titanium and its alloys may be used to fabricate multi-unit implant-supported prosthesis along with tooth-supported fixed partial dentures. The standard casting techniques involving a chemical action between metal alloys, investment materials, mechanical properties, and dimensional accuracy can be avoided through the utilization of CAD/CAM systems. The CAD/CAM restorations keep the initial properties of the beginning materials as it prepares the restoration with great accuracy using homogenous and standardized building blocks.

Several common CAD/CAM systems by GC corporation are well known as the Adava and GN1. While NobelProcera by Nobel Biocare AB, Gothenburg, Sweden is used for the fabrication of a one-piece bridge from titanium–aluminum–vanadium (Ti–6Al–4V) alloy block and it uses commercially pure titanium blocks.Appropriate resin composites have to be used on metal preparation to satisfy the demands of patients. In the oral condition it is very important to decrease the bond failure and adhesion with resin metal interface andcohesion within the resin. Sandblasting,oxide coating, plasma exposure, and primer application are various surface modifications examined to enhance the adhesive bonding of resin to commercially available pure titanium. Primers containing 10-methacryloxydecyl dihydrogen phosphate were recommended by some researchers as effective for chemical bonding between resin and Ti–6Al–4V alloy while others reported a silica-coating technique to possess good and bad effects regarding titanium alloy.Increasing micro- or nano-mechanical retention is an endeavor to enhance adhesive bonding.

An etching agent containing salt with oxygen acid has been evaluated as an alternate to sandblasting for metal bonding. The authors earlier reported that the combined use of alumina blasting and chemical etching with fluorides significantly inflated the bond strength of some resin-based self-curing luting agents to commercially pure metal or Ti–6Al–7Nb alloy. The intention behind this study was to measure the bond strength between a resin composite and Ti–6Al–4V alloy once the alloy surface was changed with aluminum oxide blasting, a fluoride etchant, and a phosphate primer. The hypothesis experienced that MDP primer improves the bond strength in consideration with ammonium ion fluoride etchant.

II. Materials and Methods

Table.1 is prepared to compile all the information on the materials based on titanium alloy, etching agents, primers and resin composite used in the particular study

Table 1: Material and its components

Material	Component	Manufacturer
NobelProcera Titanium	Ti ≥88.574, Al 5.7–6.3%, V 3.2–4.2%, N ≤0.1%, C ≤0.02%, H ≤0.010%, Fe ≤0.35%, O ≤0.22%	Nobel Biocare AB, Gothenburg, Sweden
Provisional (F-etch)	5wt% of distilled water with ammonium hydrogen fluoride	Wako Pure Chemical Ind. Ltd., Osaka, Japan
Estenia C&B Opaque Primer (MDP-primer)	10-methacryloxydecyl dihydrogen phosphate with methacrylate monomer	Kuraray Noritake Dental Inc., Tokyo, Japan
Meta Color Prime Art Jacket Opaque (FRC)	Triethyleneglycoldimethacrylate along with silanized milled-glass fiber, camphorquinone, and aromatic amine	Sun Medical Co. Ltd., Moriyama, Japan
Gradia Opaque OA3	Urethane dimethacrylate with micro silica powder	GC Corp., Tokyo, Japan
Gradia Dentin DA3	Organic composite filler, with urethane dimethacrylate and micro silica powder	GC Corp.

2.1 Specimen Groundwork

NobelProcera CAD/CAM system was used in the preparation of the specimen. The titanium alloy of NobelProcera was prepared in a disk of 6mmin diameter and 4mm in height. A total of 69 specimens were processed. Further groundwork of the disk was done with sandblasting and air abrasion. 600 grit silicon carbide abrasive paper was used to sandblast and microblaster MB102 with 50 um alumina (Hi-aluminas, ShofuInc, Kyoto,Japan) for 10s was used for air abrasion. The metal surface was kept at a distance of 25mm from the orifice and that had side air pressure of 45MPa.69 specimens were put into 3 groups each of 23 specimens as follows:

1. No etchant and No primer group.
2. No etchant with MDP primer group.
3. F-etch with MDP primer group.

According to the group of a specimen, the preparation is done as follows:

As mentioned above after alumina blasting the specimen, a micropipette was used to apply 5ul of F-etch liquid for 30s followed by rinsing with water for 15s and air drying for 5s. 1ul of primer was then put using a micropipette by delineating the bonding area using a double-coated tape of 50um thickness having a circular hole of 5mm diameter positioned on the specimen surface.

Jacket opaque milled fiber resin composite is filled in the bonded area that is surrounded by an acrylic ring of 4mm inside diameter, 0.5 mm height, and 0.5 mm wall thickness followed by light curing using curing apparatus for Dentcolor XS for 60s.On this acrylic ring an additional acrylic mold of 4mm inside diameter, 2mm tall, and 0.5mm thickness was placed on to which Gradia OA3 opaque resin of 0.2mm thickness was applied above Jacket Opaque resin which is then light cured for 60s. Finally veneering composite is filled in the acrylic resin and finished with light-curing for 210s. 3 groups of the specimens as mentioned above are prepared with or without Jacket opaque resin thus giving a total of 6 aggregates.

2.2 Shear Bond Test

A total 12 sets of 6 specimens prepared were equally divided into 2 parts of 6 sets each. The 6 sets of 6 specimens were tested for their shear bond strength at thermocycle-0 and thermocycle-200 respectively. At the state of thermocycle-0, the prepared specimenwas stored for 1hr at room temperature after which they were put in water at 37degree Celsius for 24hrs. Rest of the 6 sets of 6 specimens were tested at 20,000 cycles in a water bath in Rikothermocycling apparatus between 4degree Celsius to 60degree Celsius for 1min dwell time per bath. A shearing device ISO/TR11405 jig was used to evaluate the shear bond strength by applying shear load parallel to the bonded specimen surface in an AGS10KNG mechanical testing machine at 0.5 mm/min crosshead speed.

To analyze the bond strength, mean, and standard deviation followed by 2-way analysis of the data by ANOVA method and then comparing the mean values using post hoc Turkey compromise test, keeping the statistical significance set at 0.05 following 1-way ANOVA. An optical microscope was used to test the adhesive failure.

Failure mode at 20X magnification is categorized as follows:

1. At resin composite titanium interface (adhesive failure)
2. Within the resin composite (cohesive failure)
3. Combination of the two (combination failure)

2.3. Sem Observation

A Micro-photographic evaluation was done on two Ti-6AL-4V specimens, one experimental group and one control. The two specimens were prepared as follows:

1. One was etched and air abraded with f-etch and alumina respectively followed by a sputter of Ion coated IB3 gold.
2. Theother wasetched with F-etch for an additional 30s followed by alumina blasting. 60 minutes after the etched specimen is coated with composite resin the specimen was

cut at a right angle to the bonded interface. The second specimen was then further prepared like the first one and then the adhesive interface area was observed under 5000x magnification using a lepton microscope.

III. Result

Table 2shows the result of analysis of variance by ANOVA method corresponding to Table 3, Table 4. (Table 3) depicts shear bond strength and failure mode respectively at thermocycle-0. The Fiber milled resin composite, No-etching and MDP-primer, Fiber milled resin composite, F-etch and MDP-primer, resin composite without fiber milling, No-etching, and MDP-primer, resin composite without fiber milling, F-etch and MDP-primer groups showed significantly higher bond strengths while the fiber resin composite, No-etching and no-primer group and composite resin without fiber milling, No-etching and No-primer groups showed comparatively low bond strength values.

Failure mode is as follows, the groupfiber milled resin composite,No-etching, No-primer,fiber milled resin composite with F-etch, and MDP-primer showed only adhesive failure at the resin composite-titanium alloy interface. All specimens of composite resin without fiber milling, etching and MDP-primer exhibited failure at the resin composite-titanium alloy interface and within the resin composite. The fiber milled resin composite with No-etching but with MDP-primer and composite resin without fiber milling, No-etching and No-primer groups showed both adhesive and combination failure.

Table 2.: Results of analysis of variance that shows ANOVA results for shear bond strength

Source of variation	difference	Mean square	Sum of the squares	*F*-value	*P*-value
ANOVA corresponding to Table 3					
Surface treatment (ST)	1	3.8	3.8	0.6	0.3
Resin composite (RC)	2	457.2	901.3	76.7	0.0001
ST/RC	2	23.3	47.2	4.3	0.02
Residual	30	17.7	175.7		
ANOVA corresponding to 4					
Surface treatment (ST)	1	667.1	667.1	92.7	0.0001
Resin composite (RC)	2	739.3	1863.3	120.3	0.0001
ST/RC	2	17.3	33.3	2.3	0.1
Residual	30	7.9	224.3		

Table 3. Data on shear bond strength and failure mode analyzed at thermocycle-0.

Specimen group	Mean (SD)* (MPa)	Failure mode** (Number of specimens)
FRC/No-etching/No-primer	11.6 (2.2) [a]	Adhesive failure
FRC/No-etching/MDP-primer	26.3 (1.8) [b]	Adhesive and Combination failure
FRC/F-etch/MDP-primer	21.2 (2.1) [b]	Adhesive failure
No-FRC/No-etching/No-primer	15.0 (3.1) [a]	Adhesive and Combination failure
No-FRC/No-etching/MDP-primer	21.9 (2.1) [b]	Combination failure
No-FRC/F-etch/MDP-primer	26.2(2.8) [b]	Combination failure

The values are not statistically different as indicated by the identical small letters. ($p > 0.05$).

After 20,000 thermocycles, the mean bond strength ranged from 2.7 to 27.2 MPa (Table 4). The resin composite without fiber milling, with F-etch and MDP-primer group, exhibited the highest bond strength (27.2 MPa), in succession with composite resin without fiber milling, No-etching, and MDP-primer, Fiber milled resin composite, F-etch, and MDP-primer, and fiber resin composite, No-etching, and MDP-primer, resin composite without fiber milling, No-etching, and No-primer, and fiber milled resin composite with No-etching, and No-primer resulted in the lowest values (2.7 MPa). The failure mode in fiber resin composite with no-etching and No-primer, Fiber resin composite, No-etching, and MDP-primer, Fiber resin composite, F-etch, and MDP-primer, and composite resin without fire milling, No-etching, and No-primer group was an only adhesive failure at resin metal interface.

In contrast, all specimens of the resin composite without fiber milling, No-etching and MDP-primer and composite resin without fiber milling with F-etch and MDP-primer groups were observed to fail in the combination of failure at resin metal interface and within the composite i.e adhesive and cohesive modes both respectively.

Representative titanium specimen surfaces consisting of (a) an alumina-blasted and non-etched control and (b) an alumina-blasted specimen modified with F-etch are shown in Fig. 1. Specimen (a) was scratched with the alumina particles to form relatively smooth grooves on the surface. Specimen (b) was roughened and exhibited a greater number of micro and several hundred nano pits compared to the specimen (a).

Table 4. Data on shear bond strength and failure mode analyzed at thermocycle-20,000.

Specimen group	Mean (SD)* (MPa)	Failure mode** (number of specimens)
FRC/No-etching/No-primer	2.7 (2.4) [a]	Adhesive failure
FRC/No-etching/MDP-primer	16.8 (1.8) [c]	Adhesive failure
FRC/F-etch/MDP-primer	18.9 (2.8) [c]	Adhesive failure
No-FRC/No-etching/No-primer	14.3 (3.3) [b]	Adhesive failure
No-FRC/No-etching/MDP-primer	25.2 (2.9) [d]	Combination failure
No-FRC/F-etch/MDP-primer	27.2 (2.7) [e]	Combination failure

The values are not statistically different as indicated by the identical small letters. ($p > 0.05$).

a) b)

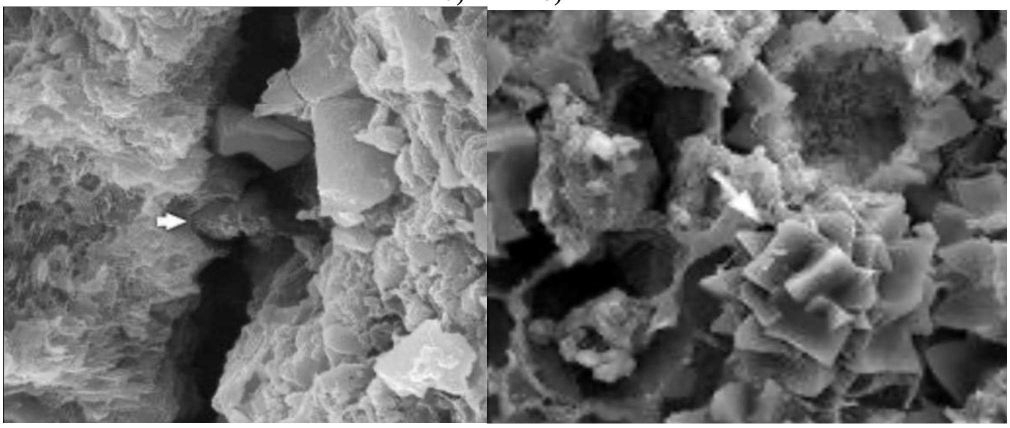

Fig. 1. Scanning electron micrographs (5000× original magnification) of Titanium alloy specimen surfaces: (a) alumina used for air abrasion ; (b) after alumina blasting, 5wt% ammonium hydrogen fluoride used for modification.

c)

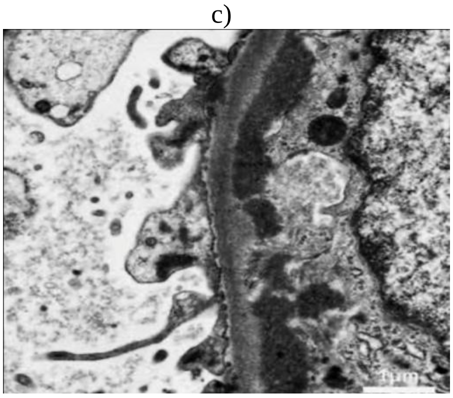

Fig. 2. Cross-sectional view of scanning electron micrograph (5000×original magnification) of the interface between titanium alloy and resin The titanium alloy of the sectioned specimen was partly dissolved with ammonium hydrogen fluoride partially dissolves the titanium alloy at the sectioned part for observation of the bonded interface

IV. Discussion

Testable hypothesis is acceptable as shown in this particular study. Previous studies have shown to be the base for the selection of concentration and etching period of ammonium hydrogen fluoride. It showed that a concentration of 5-10wt% and an etching period of 10-30s was effective to have good bond strength between titanium and resin interface. Mechanical interlocking along with the bond strength has upsurge by using alumina blasting and F-etch to roughen the titanium alloy surface. Morphology of Ti-6Al-4V alloy surface etched showed different ionization tendency and local distribution of ions according to the standard reduction potential.

Alumina-blasted forms titanium oxides, aluminum oxides, and vanadium oxides on the surface of Ti–6Al–4V alloy. This passive oxide layer may be responsible for the corrosion resistance of Ti–6Al–4V alloy. Fluoride during acid etching with ammonium hydrogen fluoride reacts with the surface of the titanium oxide layer and form titanium–fluoride compounds and replace the titanium-bound oxygen. Ti–6Al–4V alloy on the application of F-etch reacts by the formation of bubbles which is thought to occur due to the oxygen from the momentarily broken down titanium oxide. Ti–6Al–4V alloy substance can be easily ionized due to the action of ammonium hydrogen fluoride on the oxide layer. Removal of loose alumina particles may be seen as an additional effect of etching.

The SEM images in fig 1. And 2. depict that the Ti–6Al–4V alloy surface has been wetted by the primer- MDP monomer that generates numerous pits that allow diffusion of other monomers resulting in copolymerization of the diffused monomers almond with the generation of adhesive force. All this thus allows micro-mechanical retention and so-called nano-mechanical retention to be achieved. A Casting ceramic (Dicor, Densply International Inc., York, USA) usually utilizes 10wt% of ammonium hydrogen fluoride, F-etch used in the particular study has components similar to it. Also F-etch is generally used by the clinicians and doctors in the dental lab due to the deleterious effect of ammonium hydrogen fluoride in the oral environment.

Fiber milled resin composite is considered to be a flexible material and it was thought that it will relieve stress concentration at the interface. It also has lower Young's modulus as compared to the conventional resin composite and is used to fabricate coping of jacket crown, authentically. Fiber milled resin composite however showed no such benefit as a metal opaque. It could be due to the large difference between the thermal expansion coefficients of the indigenous material.

Fiber milled resin composite and Ti–6Al–4V alloy is shown to have a larger discrepancy of thermal expansion coefficients than that of the Gradia composites used as seen in this particular experimental condition in groups like fiber milled composite resin, with no etching, and primer and resin composite without fiber killing with no etching and primer, before and after thermocycling. The Actual bonding durability of the material should be confirmed properly by the clinicians as adhesive bonding is also affected by various other stresses like load, wear, and hydrolysis apart from thermal stresses in the atmosphere of the oral cavity.

V. Conclusion

The maximum bond strengths between Ti–6Al–4V alloy and the veneering resin composite was obtained when Ti–6Al–4V alloy was treated with alumina blasting, an etchant containing 5wt% ammonium hydrogen fluoride, and a phosphate primer successively. Additional use of milled-fiber resin composite as a metal opaque did not improve the bonding.

Funding
None.
Competing interests
None declared.
Ethical approval
Not required.

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Yash desai,etal. “CAD/CAM restorations and its effect on bonding composite to Ti-6Al-4V alloy.” IOSR Journal of Dental and Medical Sciences (IOSR-JDMS), 19(5), 2020, pp. 17-23.