

# Effect of Preoxidation on the Bond Strength of Titanium and Porcelain

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**Abstract** - The purpose of this study was to investigate the effect of preoxidation on porcelain titanium- bond strength and the effect of paste bonder (adhesive) on the titanium porcelain bond strength. 11 specimens of commercially pure titanium (26 × 7 × 3 mm) were prepared by different heat treatments in programmable dental furnace. Identification of the oxides formed on the metal surface was conducted with an X-Ray diffractometer with CuK $\alpha$  radiation. Vickers hardness numbers were determined. Additional 50 specimens of commercially pure titanium were used to bond with low fusing porcelain. The bond strength was measured in a universal testing machine. X-ray diffraction analysis of the surface of pure titanium revealed that the relative peak intensity of  $\alpha$ -Ti decreased and that of TiO<sub>2</sub> increased with increasing firing temperature. The Vickers hardness number decreased initially as the temperature increased but it increased remarkably above 900°C & was harder in air than vacuum. The tensile shear bond strength was highest in the green stage i.e. without preoxidation of metal, and decreased above 900°C, and was the lowest in the group without paste bonder application. The difference in bond strengths was statistically highly significant for all groups. Preoxidation under vacuum before porcelain firing can effectively improve bonding. The adhesive provided with the low fusing porcelain helps in the bond between titanium & porcelain

KEYWORDS: Titanium, Porcelain, Bond strength, Preoxidation

## INTRODUCTION

The alloys used for metal ceramic restoration can be classified as noble and base metal alloys. The high noble metal content alloys were the first group used for metal - ceramic restorations. However, noble alloys have been gradually replaced by base metal alloys due to their high cost and low sag resistance. Although base metal alloys have many superior mechanical properties, most have disadvantages such as poor biocompatibility, low tarnish and corrosion resistance, and discoloration of porcelain<sup>1,2</sup>. Titanium has several advantages such as excellent biocompatibility, high corrosion resistance, low specific gravity and low cost<sup>3</sup>. However, many practical problems with titanium for prosthodontic applications remain to be solved. High melting point and the violent chemical reactivity at higher temperatures create difficulty for both casting and porcelain bonding<sup>4,5</sup>. So, innovative casting methods<sup>6</sup> and low fusing porcelains have been developed<sup>7,8</sup>. The bonding strength of porcelain to metal depends on the oxide layer between them.<sup>9</sup> Several secondary factors may enhance the titanium - ceramic bond strength like alteration of the titanium surface using air-borne particle abrasion, acid etching, and application of a bonding agent prior to ceramic application.<sup>17</sup>

Oxidation of a metal surface increases the bonding strength, whereas excessive oxidation decreases it<sup>9</sup>. Titanium reacts strongly with gaseous elements such as

oxygen at high temperatures, and yields an excessively thick layer of TiO<sub>2</sub>, which is considered a detriment to titanium-porcelain bonding<sup>9,17</sup>. The metal oxide layer should be a monolayer in thickness to create an effective metal-ceramic bond<sup>17</sup>. Therefore, it is essential that porcelain firing should occur below 800°C to prevent excessive oxide formation<sup>4,9</sup>. Adachi *et al*<sup>4</sup> reported that the commercially pure titanium specimens oxidized at 750°C showed a well-adhering oxide layer, 32 nm in thickness, whereas the oxide layer formed on specimens heated to 1000°C was approximately 1 mm thick, with significantly lower adherence.

The purpose of this study was to investigate the influence of various heat treatments on formation of oxides on the titanium surface and consequently its effect on the bond strength between titanium and porcelain, and also on the hardness of titanium. This study also investigated the effect of paste bonder (adhesive) on the bond strength between titanium and porcelain.

## MATERIALS AND METHODS

### Preparation of heat treated specimens:

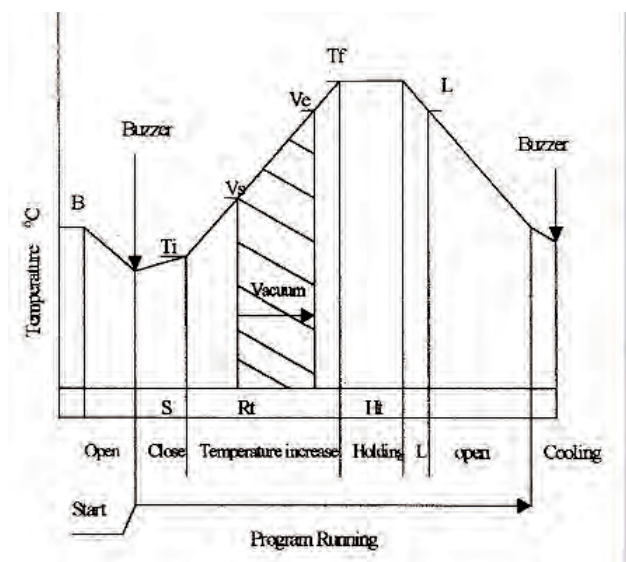
61 Commercially pure titanium specimens of size 26 x 7 x 3 mm<sup>3</sup> were used in this study (Fig. 1), which were finished with tungsten carbide bur using slow speed (approx. 15,000 rpm) and low application of pressure. The samples were ground in one direction only to remove the impurities carefully. Then all the samples were sandblasted with Aluminum oxide particles of 125 $\mu$ m at a 4-bar pressure for 10 s in a sandblaster (Unident Dental Sand Blaster, Unident Instruments Pvt. Ltd., India) and cleaned with a steam cleaner (Model no. 1F35, Inox, Omec Co.,

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**Figure 1.** Titanium specimen. Dimension – 26 mm length x 7 mm width x 3mm thickness



- B = Stand by temperature (°C)  
 S = Closing time (sec)  
 Rt = Raising temperature (°C)  
 Ti = Initial temperature (°C)  
 Tf = Final temperature (°C)  
 Vs = Vacuum 74 cm/Hg Start (°C)  
 Ve = Vacuum 74 cm/Hg End (°C)  
 Ht = Holding Time (sec)  
 L = Long term cooling (min)

**Figure 2.** The running programme of the furnace.

**Table 1.** Programs of heat treatment of Pure Titanium

Sample	Rt	Ti	Tf	Ht	Vs	Ve
1	(Cleaned with sandblaster + steam cleaner for 15 minutes)					
2	30	400	600	60	400	600
3	30	400	700	60	400	700
4	30	400	800	60	400	800
5	30	400	900	60	400	900
6	30	400	1000	60	400	1000
7	30	400	600	60	-	-
8	30	400	700	60	-	-
9	30	400	800	60	-	-
10	30	400	900	60	-	-
11	30	400	1000	60	-	-

- Rt = Raising temperature (°C)  
 Ti = Initial temperature (°C)  
 Tf = Final temperature (°C)  
 Ht = Holding Time (sec)  
 Vs = Vacuum 74 cm/Hg Start (°C)  
 Ve = Vacuum 74 cm/Hg End (°C)

Milano, Italy). The sample size was decided according to previous studies<sup>9</sup> and with respect to minimum sample size required for statistical significance. Out of these 61 samples, 11 samples were used for the X-ray diffraction analysis and microhardness test and 50 samples were used for measuring the tensile and shear bond strength of titanium-porcelain interface.

Heat treatments were carried out in a programmable dental porcelain furnace (VITA VACUMAT 500, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany) at temperatures ranging from 600 to 1000°C with either vacuum or air. The porcelain furnace had been calibrated before the study was conducted.

The schedules of heat treatments are listed in Fig. 2 and Table 1.

### X - ray diffraction analysis

Eleven titanium samples were prepared by heat treatment as shown in Table 1. A titanium sample without preoxidation, i.e. no heat treatment, was taken as the control group. The heat treatments were carried out on the other 10 samples at temperatures of 600, 700, 800, 900 and 1000 °C under vacuum and air respectively. (Figs. 3, 4) Identification of the oxides formed on the metal surface was conducted with an X-Ray diffractometer (Philips model PW 1789, Philips, Almelo, The Netherlands). The samples were subjected to X-ray diffraction, an electron beam at an accelerating voltage of 40kV directed to a copper target at a current of 25mA, at a diffraction angle  $2\theta$  in the range from 4° to 60° using  $\text{CuK}\alpha$  radiation.

### Measurement of Microhardness

The specimens used for microhardness analysis were the same as those for X-ray diffraction analysis. Vicker's hardness numbers were determined using the Vicker's microhardness test machine (UH250 Universal Hardness Tester, Reichert, Wilson Hardness, Illinois Tool Works Inc.,) at a load of 10kg and holding time of 30 seconds. The indentations were measured at 10x magnification. The diagonal length of the indentation (Fig. 6)<sup>23</sup> was measured in ocular units, from which, the Vicker's hardness number (VHN) is computed from the formula<sup>24</sup>:

$$\text{VHN} = \frac{2 P \sin(\alpha/2)}{D^2} = \frac{1.844 P}{D^2}$$

Where, P = load, kgf

D = mean diagonal of the impression, mm

$\alpha$  = face angle of the diamond  $\approx 136^\circ$

### Preparation of samples for the measurement of tensile and shear bond strength

Fifty specimens of commercially pure titanium were used to bond with low fusing porcelain (VITA Titankeramic, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany) by the sandwich technique (Fig. 6, 7) The bonding interface was 7 x 7 mm<sup>2</sup>. These specimens were already finished, sandblasted and steam cleaned.

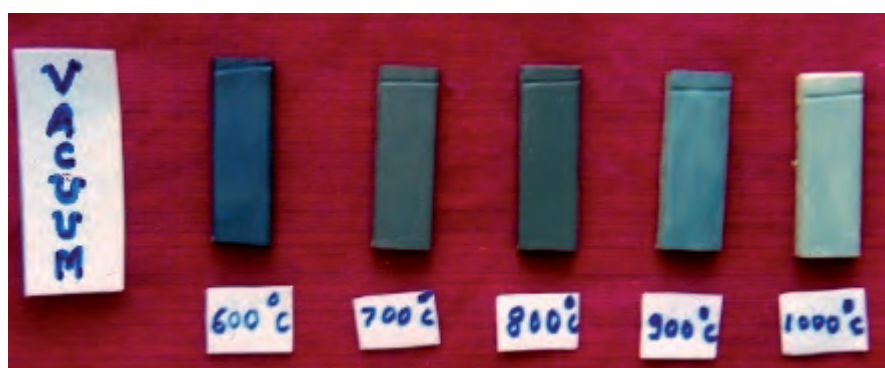


Figure 3. Preoxidation of Titanium specimens under vacuum at various temperatures



Figure 4. Preoxidation of Titanium specimens under air at various temperatures

These samples were divided equally into 5 groups (n=10):-

Group I → Control group - without preoxidation.

Group II → Preoxidation at 600°C under vacuum.

Group III → Preoxidation at 700°C under vacuum.

Group IV → Preoxidation at 800°C under vacuum.

Group V → Without preoxidation and without paste bonder (adhesive) application

VITA TITANKERAMIC low fusing porcelain was applied to the titanium samples in accordance with the actual operating sequence in the dental technology laboratory according to the firing schedule as shown in Table 2. A thin (<0.1 mm) paste bonder layer (VITA Titankeramik Paste Bonder 2200, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany – LOT. 5330 T BON) was first sintered on all the Titanium specimens except that of Group V, followed by the sintering of opaque porcelain (VITA Titankeramik VMK 95, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany – LOT. 5003) approximately 0.2 mm thick for all specimens; the final sintering of dentin porcelain (A2 shade, VITA Titankeramik Ceramique Titane, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany - LOT. 5090,) was approximately 0.8 mm thick. The thickness of the porcelain between the two titanium plates i.e. 1 mm was confirmed with an Iwanson's gauge. (Fig. 8)

#### Measurement of tension shear bond strength

All samples were tested by a modified test version of the American society for testing materials ASTM designation 2295-72 was devised to compare the complex combinations

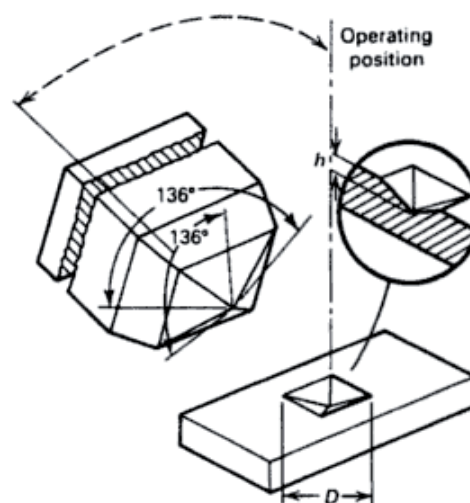


Figure 5. Diamond Pyramid indenter used for Vicker's testing and resulting indentation in the workpiece. D is the mean diagonal of the indentation in millimeters

of tensile and shear bond strength of 7 x 7 mm<sup>2</sup> interface of porcelain to titanium. The both free ends of titanium plate was held in the upper and lower grip of a Universal Testing Machine (Model 1195, Instron, Canton, MA). The upper and lower grip move away from each other to test the bond strength between porcelain and titanium at crosshead speed of 0.5mm/min resulting in tension shear force. The load required to separate the porcelain from titanium was recorded on a graph paper with a chart speed of 10mm/minute. (Fig. 9)



Figure 6. VITA TITANKERAMIC (Low fusing porcelain)



Figure 7. Titanium – Porcelain sandwich (after porcelain firing)



Figure 8. Measuring the thickness of porcelain in between two titanium specimens using Iwanson's gauge



Figure 9. Specimen held in universal testing machine (INSTRON) to detect the bond strength

Table 2. Firing schedule

	Rt	Ti	Tf	Ht	Vs	Ve
Paste bonder	60	400	600	60	400	600
Opaque	110	400	790	60	400	790
Dentin	50	400	770	60	400	770

Rt = Raising temperature (°C)

Ti = Initial temperature (°C)

Tf = Final temperature (°C)

Ht = Holding Time (sec)

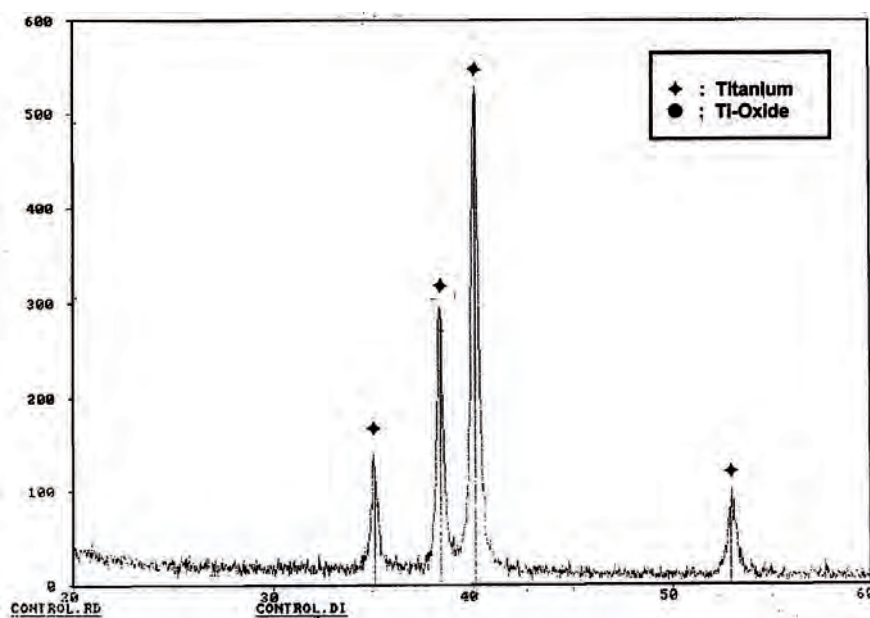
Vs = Vacuum 74 cm/Hg Start (°C)

Ve = Vacuum 74 cm/Hg End (°C)

## RESULTS

The results from the X-ray diffraction analysis of titanium treated at various temperatures in vacuum and air are shown in Graphs 1 and 2. Both patterns showed mainly single  $\alpha$ -Ti (JCPDS 00-044-1294) at initial stage. The relative intensities of  $\alpha$ -Ti decreased slightly and rutile  $\text{TiO}_2$  (JCPDS 00-021-1276) was gradually identified when the temperature increased between 700 and 800°C. The intensity of  $\alpha$ -Ti was very low while those of  $\text{TiO}_2$  increased remarkably when the temperature was raised over 900°C. No great differences were found between the relative intensities of vacuum and air treated samples.

The results of the Vickers hardness test of the heat-treated samples are shown in Graph 3. As the temperature increased, the hardness of titanium decreased initially, but it increased remarkably when the temperature increased



Graph I. X-Ray diffraction analysis of Titanium specimen - control group (without oxidation)

above 900°C. Higher values were obtained with heat treatment under air.

The mean values of the tension - shear bond strength of the porcelain titanium system of Groups I to V are given in Table 3. Titanium at the green stage exhibited the highest bond strength, while as the temperature of preoxidation increased, the bond strength between titanium and porcelain decreased. Also the bond strength between titanium and porcelain in group V i.e. without the application of paste bonder was very low as compared to group I.

## STATISTICAL ANALYSIS

The mean shear strengths of the individual groups (Group I – V) is given in Table 3.

A hypothesis of null ( $H_0$ ) was assumed that there is no significant difference between the means of the two groups. Tabulated values of 't' at 5% and 1% the level of significance for  $(n_1 + n_2 - 2)$  degree of freedom was recorded from statistical table of 't'

In the present study, to compare Groups I, II, III, IV according to preoxidation temperature and Group I and V according to adhesive bonding, the degree of freedom is 8 giving a tabulated value of

$$t_{0.05} = 2.31$$

$$t_{0.01} = 3.36$$

$$t_{0.001} = 5.04$$

The student 't' test has been applied to the data given in Table 3, to statistically compare the results of two groups. The results of the test, given in Table 4, show that difference in bond strength was statistically highly significant for all groups at 0.1% level of significance. The difference in Group III and Group IV was statistically significant at 1% level of significance.

## DISCUSSION

In contrast to most noble metal alloys that demand pre-oxidation for successful metal-ceramic adhesions<sup>21</sup>, the highest bond strengths for CPTi-ceramic system were achieved by avoiding the pre-oxidation treatment of the Ti surfaces<sup>9</sup>. Moreover, while conventional metal-ceramic systems require high fusing temperatures to enhance needed reactions, the retardation of reactions under lower temperatures seems to be successful for the Ti-ceramic system<sup>3,9</sup>.

Table 3. Mean bond strength (in kilograms ad kg / cm<sup>2</sup>) and standard deviation of different groups

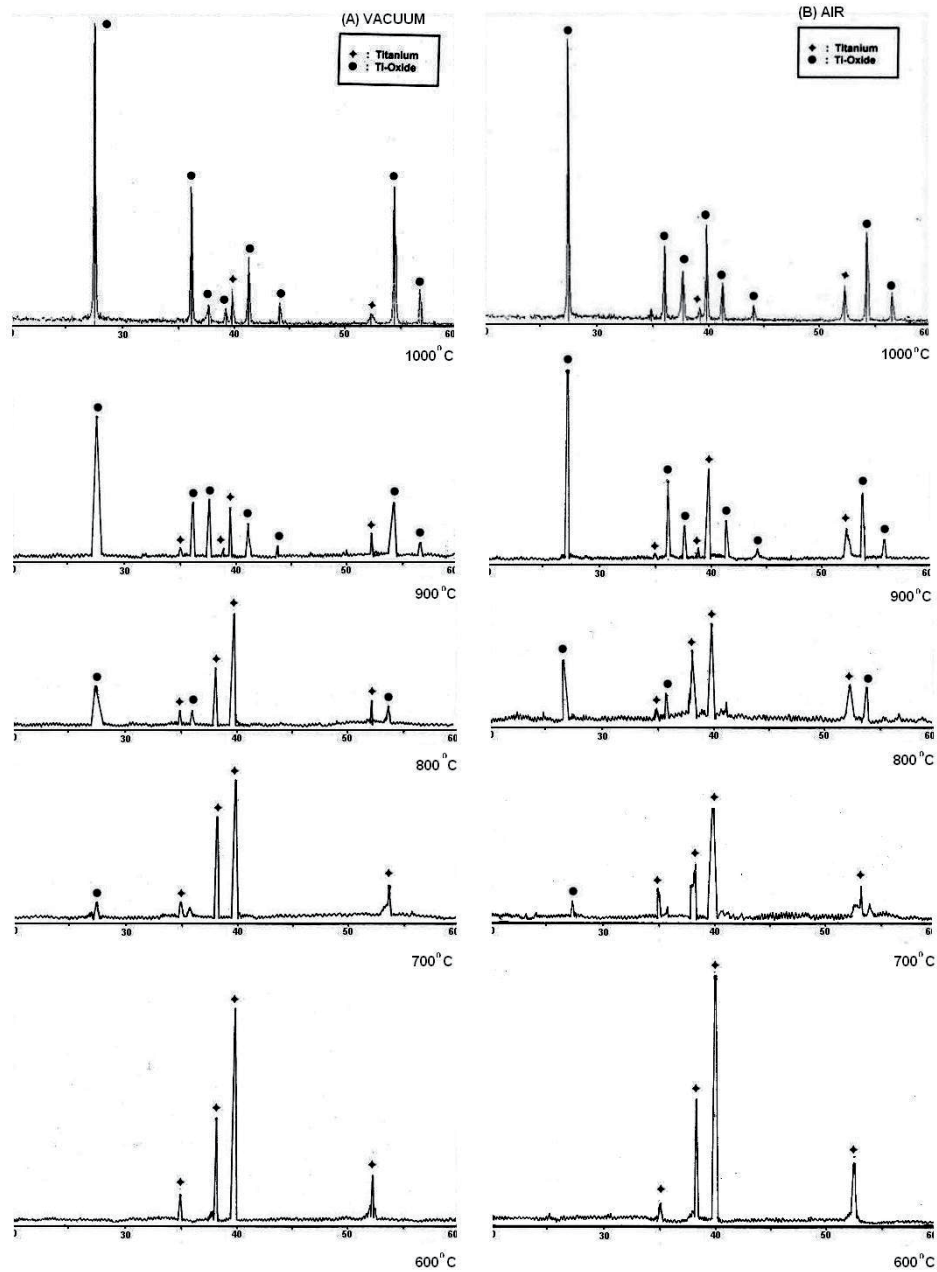
Groups	Mean bond strength ( $\bar{x} \pm S.D.$ )	
	Kilograms (kg)	Kg / cm <sup>2</sup>
I	36.20 $\pm$ 4.23	73.87 $\pm$ 8.63
II	21.58 $\pm$ 1.64	44.04 $\pm$ 3.32
III	17.16 $\pm$ 0.50	35.02 $\pm$ 1.00
IV	11.64 $\pm$ 2.43	23.75 $\pm$ 4.95
V	5.36 $\pm$ 1.44	10.93 $\pm$ 2.91

Table 4. 't' test comparison of the results of different groups of specimens

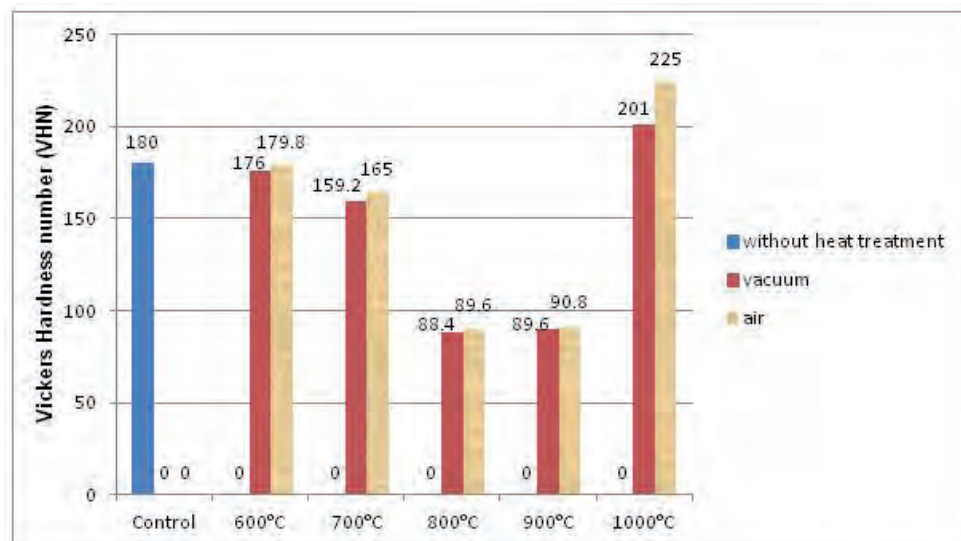
Comparison	Degree of freedom	Calculated 't' values	p values
I vs II	8	7.20	p < 0.001
I vs III	8	10.03	p < 0.001
I vs IV	8	11.27	p < 0.001
II vs III	8	5.90	p < 0.001
II vs IV	8	7.59	p < 0.001
III vs IV	8	4.97	p < 0.01 *
I vs V	8	15.43	p < 0.001

\*p < 0.01 – Significant

p < 0.001 – Highly significant



Graph 2. X-Ray diffraction analysis of Titanium specimen – preoxidised in vacuum (A) and air (B)



Graph 3. Effect of heat treatment on the hardness of titanium

The principal problem in bonding ceramics to Ti is the extensive dissolution of oxygen into the Ti lattice, resulting in thick, oxygen-rich, Ti layer. This produces the so-called "α-case", which impairs the mechanical compatibility of the Ti-ceramic interface<sup>22</sup>.

X-ray diffraction analysis revealed that titanium treated at various temperatures in vacuum and air showed mainly single phase at the initial stage. The relative intensity of α-Ti decreased slightly and TiO<sub>2</sub> was gradually identified when temperature increased between 700°C & 800°C. The intensity of α-Ti was very low while those of TiO<sub>2</sub> increased remarkably when the temperature was raised over 900°C. Lower oxide adherence strength was observed for specimens at higher temperatures, especially at 1000°C. In these specimens, peeling of the oxide layer was observed. The results were consistent with the reported oxidation behavior of titanium<sup>4,9</sup>. The optimum firing temperature is judged to be 700°C to 750°C for porcelain veneering of titanium<sup>4, 18, 19, 20</sup>. Hence, preoxidation temperatures for this study were selected ranging from 600°C to 1000°C.

The metal to which porcelain is fused should be as hard as possible to prevent firing deformation. In this study, to measure the hardness of titanium, Vickers hardness test was used because according to ADA specifications, this test proved to give more standard satisfactory values compared to other Brinell, Rockwell and Knoop hardness tests. The results of the Vickers hardness test showed that the hardness of titanium initially decreased as the temperature increased. But it increased remarkably when the preoxidation temperature was raised over 900°C and hardness of titanium was more in air than in vacuum. One of the reasons for initial decrease in hardness with the increase in temperature may be 'annealing'. As we had carried out the study on wrought titanium samples, the effect of strain hardening could be reversed by simply heating the metal. At temperatures greater than 900°C, the hardness increased because after the completion of the annealing procedure, the oxides penetrate inside the metal and this causes an increase in the hardness of titanium.

The theory of ceramic-to-metal bonding has focused on four possible mechanisms: (1) van der Waals's forces, (2) compressive forces generated in thermal expansion, (3) mechanical bonding with surface geometry, and (4) chemical bonding with the oxides in the ceramic and the metal<sup>10</sup>. Various in vitro test methods have been designed and selected by researchers to measure the bond strength at the ceramic-metal interface and to understand the bonding roles of the four mechanisms<sup>10</sup>, which can be classified according to the nature of stresses created such as shear, tension, combination of shear and tension, flexure, and torsion test designs<sup>11</sup>. However, accurate measurement of the bond strength at the metal-ceramic interface poses several questions, as the complexity of the bonding mechanism currently defies the development of an ideal test design<sup>11, 12</sup>.

Characteristics of an ideal test of the metal-ceramic bond strength have been described in the dental literature as follow<sup>13, 14</sup>:

1. The testing configuration induces adhesive metal-ceramic failure within the interfacial adherence zone, but not cohesive failure within the porcelain.

2. Specimen loading generates uniform stress distribution along the metal-ceramic interface, permitting accurate force/area bond strength calculations.

3. Specimen preparation and testing procedures are uncomplicated, allowing evaluation of many metal-ceramic combinations.

Shear tests, which are simple to perform, are the most popular, and several metal-ceramic bond tests have been done in this mode with different test configuration designs. The planar shear test is claimed to be a well-suited design for evaluation of metal-ceramic. In the flexural (bend) tests, i.e. 3- and 4-point bending tests, porcelain was bonded to the tension surface of a metal bar only in the middle part. However, the result in all of these studies is quoted as the critical stress at bond failure. In an attempt to compare the actual characteristics of the stress distribution in different bond tests, finite element stress analysis has been reported, which have been compared with the experimental observations of fracture location and type of failure. In the 3- and 4-point bending tests there is a severe stress concentration at the porcelain endpoints. The stress distribution in shear tests shown in the finite element stress analysis resulted from a force ideally applied at the metal-porcelain interface, which does not occur experimentally. For this reason, the failure of the bonded system may possibly be initiated within the porcelain, close to the interface but not on it, in which case the failure is caused by tensile stresses<sup>15</sup>.

Hence, in this study, a combination shear and tension test was used to test the bond between titanium and low fusing porcelain. This tension shear test adopted from the American society for testing material (ASTM) test, version D-2295 - 72 was developed by Wight *et al*<sup>11, 16</sup>. In this test, two parallel metal samples separated by a block of porcelain were used, which closely simulated the actual titanium- porcelain bond interface. The advantage of this test was that the forces were not primarily directed at metal-ceramic interfaces because the two bonding interfaces that were not aligned at the middle of the thickness of each specimen, but instead, test loading was directed diagonally for development of a combination of shear and tensile stresses to simulate complex, clinical stress situations<sup>11</sup>. A flat surface was used in this test to eliminate additional positive and negative stress components which occurred on a curved surface resulting from different coefficients of thermal expansion. As Adachi *et al*<sup>4</sup> showed that oxide adherence to metal surface at higher temperature is lower, so the authors did not consider 900°C and 1000°C samples in bond strength studies. The bond strength results revealed that it is highest in the green stage i.e. without preoxidation. As the temperature increased, the bond strength between titanium & porcelain decreased.

Bond strength & X-ray diffraction analysis results showed that as the temperature increased, the α-Ti phase decreased and titanium oxides increased. So, an increase in temperature causes increase in TiO<sub>2</sub> on the titanium surface along with a decrease in the titanium porcelain bond strength. This shows that TiO<sub>2</sub> is a weak phase and it causes a decrease in the bond strength of titanium - porcelain. The results of the Vickers hardness test showed that titanium increases in hardness after a suitable heat treatment, especially over 900°C. However, the formation of an oxide layer on the titanium surface was usually noted with tem-

peratures over 900°C, producing an extra-hardness but a drastic decrease in the titanium porcelain bond strength. In conclusion, a decrease in the oxidation of titanium can suppress the formation of a weak phase, thus increasing the porcelain titanium bond strength.

According to the claims of the manufacturer, “*the VITA BONDER enabled increasing the adhesive bond strength of titanium and low fusing porcelain. The bonding of titanium oxides during firing procedures in the bonder matrix leads to a secure and durable adhesive bond. The titanium/ceramic adhesive bond is equal to conventional systems.*” Hence, this study also included the evaluation of the effectiveness of the bonding agent (paste bonder) provided along with the low fusing porcelain. The bond strength of group V i.e. without preoxidation & without paste bonder application was much lesser as compared to that of group I i.e. without preoxidation & with paste bonder application.

From the results of this study, it could be said that for the success of the porcelain - titanium system, it is mandatory to demand two elementary properties i.e. the hardness of titanium to prevent firing deformation and a good titanium ceramic bond. The preoxidation of titanium increases its hardness, but consequently causes a dramatic decrease in the bond strength. Hence it is essential to optimize the preoxidation temperature in such a way that the desired bond strength is achieved along with an optimum hardness of titanium. .

## CONCLUSION

The effects of heat treatment on the porcelain - titanium interface reactions and bond strength were investigated. From the results of this study, the following can be stated:-

1. The X-ray diffraction analysis revealed that the relative intensity of the  $\alpha$ -Ti decreased, while that of the TiO<sub>2</sub> increased when the preoxidation temperature was raised.
2. The tension shear bond strength of the porcelain - titanium system was the highest in the group without preoxidation.
3. Vickers hardness test showed that initially as the temperature increased, the hardness of titanium decreased but it increased remarkably above 900°C
4. The tension shear bond strength without the application of paste bonder was the lowest among all groups.

Hence, the clinical implication of this study is that for any titanium – porcelain prostheses, to have maximum bond strength between titanium and porcelain, always use a low-fusing porcelain so that the firing temperature of porcelain will be below 700 – 750 °C and avoid conventional preoxidation of metal in case of titanium.

## ADDRESS FOR CORRESPONDENCE

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