

Effect of Al₂O₃ Sandblasting and Silicoating on Bond Strength of a Resin Cement to Titanium Implant

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Al₂O₃ sandblasting과 Silicoating이 titanium과 레진시멘트의 접착강도에 미치는 영향

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[요약]

연구 목적: 임플란트의 하부구조를 상부 구조와 연결하는 레진시멘트의 접착강도를 높이기 위한 기계적 화학적인 표면 처리 방법들이 연구 되고 있다. 이 연구에서 다양한 크기의 Al₂O₃ sandblasting과 Silano Pen으로 표면처리한 티타늄과 레진 시멘트의 접착강도를 알아보고자 한다.

연구 방법: 12개의 티타늄(Ti-6Al-4V)시편을 디스크 형태로 제작하여 자가중합 수지에 매립하였다. 이들을 각각 6개의 군으로 나누어 50µm, 90µm, 110µm 등 3가지 크기의 Al₂O₃로 sandblasting 하는 조건과 Al₂O₃로 sandblasting한 후 Silano Pen(Bredent, bredent GmbH & Co.KG, Senden, Germany)을 사용한 군으로 나누었다. 표면처리 한 티타늄 표면에 레진시멘트(Duolink dual syringe, Bisco, USA)으로 접착하였다. 그 후 증류수(37°C)에 24시간 보관 후 접착강도 실험을 시행하였고, SEM을 사용하여 표면처리 한 표면과 접착강도 실험 후 파절양상을 관찰하였다.

결과: 통계학적 분석에 따르면 Silano Pen을 사용하여 표면처리한 군들의 접착강도가 높았다(P<0.05).

결론: Silano Pen을 사용하는 것이 티타늄과 레진시멘트의 접착강도를 증가 시킨다.

주제어 : Al₂O₃ sandblasting, silicoating, titanium, bond strength

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I. INTRODUCTION

Dental implants are now becoming a more popular treatment to replace missing teeth, as they provide a longer-term solution, slow down bone loss and preserve nearby healthy tooth tissue. Implants are made from titanium, a material that is well tolerated by bone and integrates easily with bone tissue (Brauner, 1993; Okabe et al, 1995; Scarano et al, 2003). Titanium has many advantages as a prosthesis material, including excellent biocompatibility, high strength to weight ratio, low density, sufficient corrosion resistance, and low cost compared to noble alloys (Parr et al, 1985; Zavanelli et al, 2000). However there are some disadvantages and potential problems that might arise after treatment is completed. Lately, titanium implants have shown bonding problems with resin cements. If there is a separation between titanium and resin, cracks or crazing in that area may be a nidus for microorganisms and plaque to accumulate, possibly resulting in adhesive bond failures. Many different surface treatments have been proposed to improve the strength of this bonded interface. These treatments include sandblasting, silicoating, using functional monomers, acid etching, and many others. Studies have shown the treatments have been effective at increasing bond strength, albeit at varied amounts (May et al, 1995; Koizumi et al, 2006; Ban et al, 2006).

Many bonding systems are commercially available and each manufacturer touts better bond strength with their system. One of these systems is the Rocatec system by 3M ESPE (Seefeld, Germany). Rocatec was introduced to the German market in 1989 with advantages over the classic silicoater process in the heat-free generation of the silicate

layer and its visual monitoring on metal. The manufacturer states that the system is compatible for use with all metals used in dentistry including titanium. Rocatec is a 2 tribochemical method for silicating surfaces. Tribochemistry involves creating chemical bonds by applying mechanical energy. This supply of energy may take the form of rubbing, grinding, or sandblasting. There is no application of heat or light which would normally be the case with chemical reactions.

Another system that is commercially available is Silano Pen by Bredent (Senden, Germany). The manufacturer claims that the Silano Pen bonding system allows preparation of a chemical-micromechanical bond between implants and resin cements. In this system, the creation of highly stable and durable bonding

between implants and resin cements is based on the combination of a special gas mixture 3 with a bonding liquid. The special gas mixture is processed with the firing device known as the Silano Pen. Short firing with the Silano Pen results in fine cleaning and, simultaneously, silicate formation and activation of the surface to be processed. The ensuing application of the bonding agent optimizes the bond.

The Silano Pen performed significantly better with the base metal alloy (Co-Cr) than either spark erosion or Rocatec (Janda et al, 2007). Its performance on the gold alloy and titanium was similar to both of the other bonding systems. Silano Pen is a relatively easy system to use, but it is not well documented in the literature.

The purpose of this study was to evaluate the bond strength between titanium implants and resin cements using different surface treatments including treatment with Silano Pen.

II. METHODS

Ti6Al4V disks, 15 mm in diameter and 2mm in thickness were embedded in polyethylene molds using polymethyl methacrylate (Self Curing, Vertex, Netherlands), with one side of the disk exposed for cement bonding. They were polished with 800-grit silicone carbide abrasive under water cooling, and then ultrasonically cleaned in distilled water for three minutes. Subsequently, the disks

were randomly divided into six groups according to the surface conditioning method to be applied (N=60, n=10 per group). Group 1, 2, 3 were blasted with 50, 90 and 110 μ m Al₂O₃. The other groups, called the Silano Pen(SP), had their surfaces blasted with 50, 90 and 110 μ m Al₂O₃ and treated with Silano Pen.

〈Table 1〉 shows the batch numbers, and manufacturers of the titanium, aluminum oxide, Silano Pen, and resin cement used in this study.

Table 1. Materials used in this study

Product	Batch Number	Manufacturer/Supplier
Ti-6Al-4V (Accu-thermII-1000)	8952315	J F Jelenco & Co, NY, USA
Aluminum oxide (Al ₂ O ₃ , Strahlmittel)	50 μ m-1147453, 90 μ m-13239, 110 μ m- 994495	Renfert GmbH, Hilzingen, Germany
Silano Pen	305727	Bredent GmbH, Senden, Germany
Resin cement(Duo-Link)	1100011834	BISCO dental products, BC, CANADA

1. Surface conditioning methods

The following surface conditioning methods were employed per experimental group.

Group 1, 2, 3 were performed 50, 90, 110- μ m aluminum oxide using an air abrasion device. The nozzle was held perpendicular to the surface from a distance of approximately 10mm for 15sec/cm² at a pressure of 2.5 bar. The substrate surface was rinsed. Group 4, 5, 6 were performed with 50, 90, 110- μ m aluminum oxide using an air abrasion device. The nozzle was held perpendicular to the surface from a distance of approximately 10mm for 15sec/cm² at a pressure of 2.5 bar. The substrate surface was rinsed for 20 seconds and air-dried for five seconds. Heat treatment was achieved by applying the flame of Silano Pen (Bredent, Senden, Germany) for 5 sec/cm² at the surface. The surface

was then left to cool down at room temperature. Following which, the corresponding silane (Haftvermittler, Bredent) was applied to the surface with a disposable brush and left for three minutes for its reaction to be completed.

2. Surface roughness analysis

Surface roughness was measured by a profilometer (Surftest SV.400, Mitutoyo Co, Kanagawa, Japan) equipped with a diamond-tracing stylus. Mean roughness Ra was calculated.

3. Bonding procedure

A dual-cure resin cement (Duo-Link, Bisco Inc., Hilzingen, Germany) was applied to make cement blocks (10 cylinder shape resin cements on each titanium specimen) on the specimens embedded in

translucent polyethylene molds. The specimens were light-polymerized for 30 seconds. All specimens were kept at 37°C for 24 hours.

4. Bond test

The specimens were mounted in the jig of a universal testing machine, and load was applied to the adhesive interface until failure occurred (crosshead speed: 1.0mm/min). The maximum force to produce failure was recorded (MPa) using a corresponding software.

5. Statistical analysis

Kruskal-Wallis test was applied. And Mann-Whitney test was used to identify significant difference. In all the statistical analyses the level of significance was set at $\alpha = 0.05$. P values less than 0.05 were considered to be statistically significant in all tests.

6. Scanning electron microscope (SEM) evaluation

Titanium surfaces treated with 110 μ m Al₂O₃ and Silano Pen before and after bond test were submitted for SEM evaluation to qualitatively analyze the surface to be bonded after the various surface treatments. The distributions of failure patterns were compared among the groups. Magnifications of 1000 and 1500 \times were used.

III. RESULTS

Mann-Whitney test revealed the existence of significant between-group differences(Fig. 1). The

roughness tests of 50 μ m had significantly lower than 90, 110 μ m ($p < 0.05$). Silano Pen treatment groups had significantly lower roughness compared to those of untreated groups ($p < 0.05$). The roughness values for 50, 90, 110 μ m were 1.3, 1.90, 2.04 μ m, and for the Silano Pen treatment groups were 0.64 (50 μ m), 1.21 (90 μ m), 1.23 (110 μ m) μ m, showing that Silano Pen treatment groups significantly reduced roughness values in this study.

Result of bond strength tests are given in <Fig. 2> According to the test, Silano Pen treatment groups produced a significant increase in the bond strength in comparison with sandblasted specimens ($p < 0.05$). The bond strength values for 50, 90, 110 μ m were 16.80, 20.30, 20.57MPa, and for the Silano Pen treatment groups were 45.45(50 μ m), 53.69(90 μ m), 55.31(110 μ m)MPa, showing that Silano Pen treatment groups significantly increased bond strength values. The bond strength tests of 50 μ m showed significantly lower than 90, 110 μ m ($p < 0.05$).

SEM images are reported in <Fig. 3>. Compared to the Silano Pen groups, sandblasted specimens presented with much greater surface roughness (Fig. 3). Residual alumina on the surface of titanium was observed. Sandblasted specimens mainly failed adhesively at the titanium interface (Fig. 4 c, e). Although surface irregularities were evident, a small amount of remaining resin cement could be detected on the titanium surface after load. In Silano Pen groups mixed failures were prevalent(Fig. 4 d, f). Resin cement remnants retained over a treated titanium were seen in SEM images of specimens from Silano Pen groups.

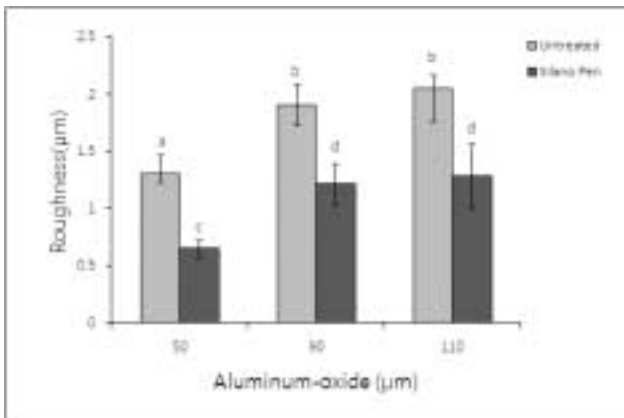


Fig. 1. Roughness of 3 different aluminum oxides(50, 90, 110µm). Different letters mean significant difference at P<0.05 level

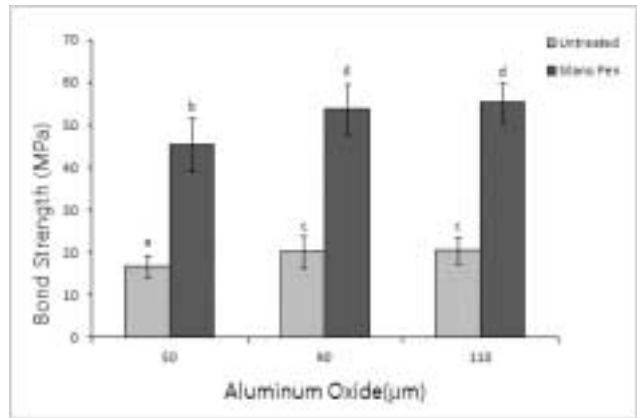


Fig. 2. Bond strength of 3 different aluminum oxides(50, 90, 110µm). Different letters mean significant difference at P<0.05 level

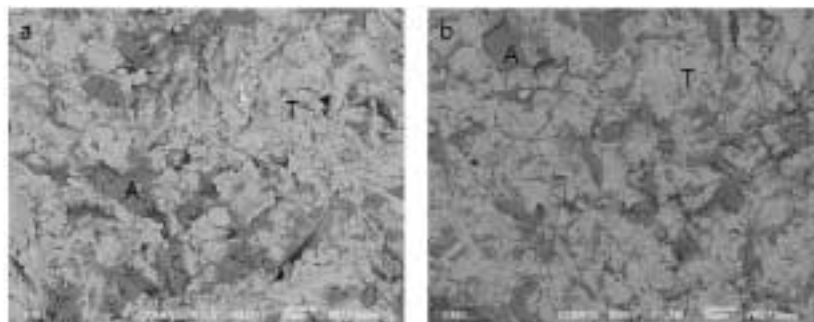


Fig. 3. Titanium surfaces treated with 110µm Al₂O₃(a) and Silano Per(b) viewed with SEM under 1000 X magnifications. T: Titanium, A: Alumina

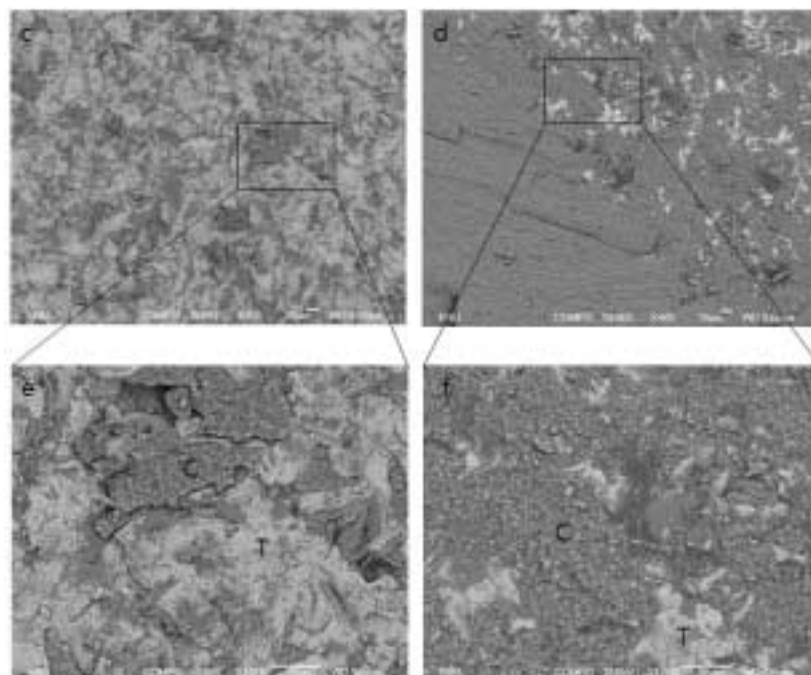


Fig. 4. SEM images (400 X, 1000 X) of titanium surfaces after bond test. Images show disks treated with 110µm Al₂O₃ (c,e) and Silano Per(d,f). C: Resin cement, T: Titanium

IV. DISCUSSION

Many surface treatments have been proposed in the literature to increase the bond strength between metal and resin cement. These treatments include surface roughening to provide micromechanical retention, chemical bonding between the resin cement and titanium, or treatments that combine both a roughening and a chemical component. The effects of sandblasting or air-particle abrasion on the bond strength between resin cement and titanium have been demonstrated in the literature (Giachetti, 2004). Research showed an increase in the bond strength after sandblasting.

In this study, the roughness tests of 50 μ m had significantly lower than 90, 110 μ m. Surface roughening by grit-blasting with abrasives Alumina (Al₂O₃) depends on the size and shape of the abrasive. Silano Pen treatment groups had significantly lower roughness compared to those of untreated groups. The special gas mixture is processed with the firing device known as the Silano Pen. Short firing with the Silano Pen results in fine cleaning and, simultaneously, silicate formation and activation of the surface to be processed. The ensuing application of the bonding agent optimizes the bond. All these steps effected the roughness.

Systems that are based on silica coating and silanization have been thoroughly

studied in the literature (Mukai et al, 1995). Most authors showed significantly improved bond strengths by using these systems, which include the Silicoater, Rocatec, and the Kevloc bonding system. Janda et al (2007) bonded resin to Ti with Silano Pen and reported bond strengths of 18.4MPa and 11.1MPa before and after thermocycling,

respectively. Murat et al (2010) reported that Silano Pen showed highest test results (506,02 \pm 18,04 N) and differ from sandblasting with 50 μ m Al₂O₃ on the retention of single crowns and implant abutments. The bond strength for the Silano Pen treatment groups significantly increased bond strength values in this study.

Presently, satisfactory bond strength values of resin cement to titanium implant are yet to be determined for clinically successful performance. Nonetheless, the bond values obtained for the titanium tested in this study could be considered sufficient with all the herein-evaluated surface conditioning methods.

Limitations of the study may include that it is an in vitro study and conditions that may affect the bond strength in vivo were not tested. Also, thermal stresses induced by the thermocycling were not tested, and with regard to testing methodology, it may be that specimen geometry combined with bond testing parameters used may not accurately reflect the stress state observed in an actual prosthesis during function.

V. CONCLUSION

Within the limitations of this study, it can be concluded that treating titanium surfaces with chemical procedures such as Silano Pen is beneficial for improving the titanium-resin cement bond strength.

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