



## Measuring and comparing the shear bond strength of acrylic resin veneer to nickel –chromium alloy after different metal surface treatments

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### Abstract

The bond strength of metal-resin interface of prosthesis is a key factor in determining the serviceability of that prosthesis. The main objective of this study was to determine the effect of different surface treatment on the shear bond strength of Ni-Cr alloy (CB-Blando 72 alloy) and heat cured acrylic veneer resin (Ivoclar Co.). One hundred twenty eight disc shaped samples from base plate wax were prepared, the diameter of disc was (8mm×4.5mm). After casting the metal, the samples were cleaned, finished, and polished by silicon carbide abrasive paper with a grit size (600 $\mu$ m), and then the profilometer was used to determine the surface roughness of the samples (Ra) which ranged between (0.09-0.13 $\mu$ m). Then the samples were divided into 2 categories, each category contain (64) samples which also were subdivided into 8 group, the first category include (A, B, C, D, E, F, G, H) and the second category include (A<sub>t</sub>, B<sub>t</sub>, C<sub>t</sub>, D<sub>t</sub>, E<sub>t</sub>, F<sub>t</sub>, G<sub>t</sub>, H<sub>t</sub>), according to the metal surface treatment which include :

**Group A, A<sub>t</sub>:** Received no treatment and serve as control.

**Group B, B<sub>t</sub>:** Received chemical treatment using chemical bonding agent (Metal primer II).

**Group C, C<sub>t</sub>:** Received micromechanical treatment using air abrasion with 50 $\mu$ m Al<sub>2</sub>O<sub>3</sub>.

**Group D, D<sub>t</sub>:** Received micromechanical treatment using air abrasion with 50 $\mu$ m Al<sub>2</sub>O<sub>3</sub> +metal primer II

**Group E, E<sub>t</sub>:** Received micromechanical treatment using air abrasion with 110 $\mu$ m Al<sub>2</sub>O<sub>3</sub>.

**Group F, F<sub>t</sub>:** Received micromechanical / chemical treatment using (air Abrasion 110 $\mu$ m Al<sub>2</sub>O<sub>3</sub> + metal primer II application).

**Group G, G<sub>t</sub>:** Received micromechanical treatment using air abrasion 250 $\mu$ m Al<sub>2</sub>O<sub>3</sub>.

**Group H, H<sub>t</sub>:** Received micromechanical /chemical treatment using (air abrasion 250 $\mu$ m Al<sub>2</sub>O<sub>3</sub> +metal primer II).

The acrylic was then prepared as round shape (8mm in diameter and 2mm in thickness) that was placed on the metal surface treatment; the acrylic was fabricated in the same conventional way for construction veneer resin.

For the specimens in the first category, the specimens were tested under dry condition, while in the second category; the specimens were tested after (24) hours

storage in water room temperature (37°C) and (1000 time) thermocycling. The shear bond strength was tested in instron universal testing machine.

The results before and after thermocycling showed that the air abrasion with 50µm Al<sub>2</sub>O<sub>3</sub> plus metal primer II group gave the highest shear bond values, followed by air abrasion with 110µm Al<sub>2</sub>O<sub>3</sub> plus metal primer II, followed by air abrasion with 50µm Al<sub>2</sub>O<sub>3</sub>, followed by air abrasion with 250µm Al<sub>2</sub>O<sub>3</sub> plus metal primer II, followed by air abrasion with 110µm Al<sub>2</sub>O<sub>3</sub>, followed by air abrasion with 250µm, followed by metal primer II group, and followed by untreated group.

Also after thermocycling showed that the micromechanical/chemical treatment (air abrasion with Al<sub>2</sub>O<sub>3</sub>+metal primer II) gave the highest and more durable shear bond strength values than other groups.

## Introduction

Resins of methacrylate –based polymers have been used for a long time as tooth colored veneering materials on cast crown and bridges (1,2).

The low retentive force of the resin in the metal casting might lead to the loss of veneer when it is repeatedly subjected to occlusal stress (3).

During the last years several works were accomplished on the increase of the bond strength of resin to metals, mainly through surface treatment of metallic alloys (4).

There are several methods available for bonding resin veneers to metal. Metal surface treatments have been classified according to factors that control the bonding between the metallic alloys and the acrylic resins, including mechanical, chemical, and combined mechanical chemical retentive bonding systems (5,6).

Under cut designs such as struts, loops, mesh work, and beads have been incorporate into wax framework pattern before casting. These designs mechanically enhance the attachment of the resin to the cast frameworks (7).

The major disadvantage of mechanical retention between the resin and metal is poor mechanical sealing which permit seepage of oral fluids between the resin-metal interface that leads to increased retention of microorganism and fluids,

deterioration, discoloration and subsequent unfavorable results (7,8).

Since 1980, methods have been reported to create a chemical bond between dental alloy and resins without the use of mechanical retention and there by providing more space for the veneer layers, especially in limited access areas. The chemical attachment of an opaque layer to the resin – metal interface limited micro leakage at the metal-resin interface that occurred because of polymerization shrinkage and mismatch of coefficient of thermal expansion between the resin and metal (9,10).

An optimum metal – resin bonding system would include a simple procedure producing maximum retention and high consistent bond strength values (10,11).

In the present study, metal resin shear bond strength has been evaluated by using different metal surface treatment in order to obtain better retention and prevent unfavorable results of metal-resin interface.

## Materials and Methods

The materials used in this study are listed as follows:

- Modeling base plate wax (Degussa, Lot No.300601, Exp Date:7:2010, Germany ).

- Ring liner (Kera –vlies, asbestos – free strips, Dentaraum, Germany).
- Surface tension reducing agent (nodrenta, FINO SPANN-Ex, Exp Date: 2009, Germany).
- Phosphate bonded investment (free carbon, Gilvest, Exp.Date:4:2008, Germany).
- Special liquid for mixing the investment (Gilvest,liquid,Exp. Date:4:2008, Germany)
- Nickel –chromium dental casting alloy: CB Blando 72(Harakeyma Dental MFG .CO. 1td, Japan).
- Aluminum oxide powder (polishing beads, 50 $\mu$ m, 110 $\mu$ m, 250 $\mu$ m, shera, Germany).Fig.(1:A,B,C).
- Distal water (Iraq).
- Metal primer II (MPII), (Chemical bonding agent), (GC corp, Toky, Exp. Date: 6:2008, Japan).
- Hot cure acrylic resin for crown and bridge facing (opaque and body), (liquid & powder), (Ivoclar ,Liechtenstein,Exp. Date:2007

#### Equipments and Instruments

- Glass slab (Vita, Germany).
- Copper ring no.27 (Produits Dentires Pd, Switzerland). Fig(2)
- Micrometer wax caliper device (Dentaram ,Germany).
- Metal casting ring & crucible former (size 3x, Degussa, Germany).
- Vaccum–auto mixing device with vibrator (Bego, Germany).
- Electrical furnace for wax burne (200 $^{\circ}$ c -1300 $^{\circ}$  c,Detor ,QD,England).
- Sensitive electron balance (Germany).
- Manual driven broken arm centrifugal casting machine (TS, Degusa, Germany).
- Gas-oxyen signal orifice torch (Degusa, Germany).
- Profilometer device ("Taylsuref " 4, England).Fig (3).

- Sand blast machine (Bego,Germany).
- Specimen metal holder for sandblasting (locally made).
- Ultrasonic cleaning machine (Braun, Germany).
- Ivomat (Ivoclar ,Germany ).
- Incubator (Germany).
- Thermocycling machine (Iraq).Fig. (4).
- Metal chisel for shear test (Hand made).
- Instron universal testing machine (zwick, 1195, England).
- Olympus photomicroscope system with exposure control unites (Japan).

#### Methods

##### • A-Grouping of the Sample

One hundred twenty eight metal samples were divided into two categories each category contain 64 samples and were subdivided into 8 groups according to the metal surface treatment. In the first category, the samples (A, B, C, D, E, F, G, and H groups) were tested under dry condition (without thermocycling), while in the second category the samples (A<sub>t</sub>, B<sub>t</sub>, C<sub>t</sub>, D<sub>t</sub>, E<sub>t</sub>, F<sub>t</sub>, G<sub>t</sub>, and H<sub>t</sub> groups) tested after (24 hours) storage in water at room temperature (37 $^{\circ}$ C) and (1000 time) thermocycling (with thermocycling).

##### • General Preparation of the Specimens

Three sheets of modeling wax (17.5 cm in length, 9.5 cm in width and 1.5 mm in thickness) was punched with a copper ring no. 27 (8mm in diameter) as mold Fig. (6), five points of each wax pattern could be measured by wax caliper device to check uniform thickness (4.5mm) for each pattern. (12,13).

Each 6 samples were sprued and invested in one casting ring using

Gilvest investment. The wax wire was (2.5 mm) in diameter attached to running bar of (4mm), the wax pattern was attached to the crucible former to be away from the end of the ring in about (6mm).

After completion of spruing, investing wax done immediately using casting ring size (3x), which was lined with a single layer of asbestos free ring liner. A full liner was wetted with water and adapted slightly on the inner surface of the ring. The wax samples were lubricated with surface tension reducing agent (debubblizer) and left for drying<sup>(14)</sup>. Phosphate bonded investment (Gilvest) was mixed according to the manufacturer's instruction, each (100g) of powder was mixed with (24 ml) of its special liquid without dilution, measured by using a calibrated glass measuring cylinder. The measured liquid was poured into a damp-mixing bowl followed that the powder was added. Fifteen seconds of initial hand spatulation were followed by forty five seconds of vacuum mechanical spatulation to minimize the chance of air bubbles, which could be attached to the pattern and compromise the result. A brush was used to apply the investment material gently to the wax pattern then remaining investment was poured into the ring carefully with the aid of vibrator to prevent air entrapment. Then the ring was kept a side for setting according to the investment manufacture instruction.

The burn out step was carried out according to the manufacturer's recommendation for temperatures and timing. The casting ring was kept in electrical furnace at room temperature with crucible former opening side down ward, to facilitate evacuation of the molten wax after heating<sup>(14)</sup>.

The ring was then heated gradually to (200°C) and was kept at this temperature for (30 minutes). After that temperature was elevated

gradually to the final level (950°C) and was kept at that degree for (60 minutes).

A new metal of CB-Blando 72 was used for each casting, which is beryllium-free nickel chromium alloy; the alloy was selected because of its availability and broad usage.

Manual driven broken arm centrifuged casting machine was prepared to be ready for casting. All the casting were made with the same casting pressure and to achieve that the machine was wound for four turns<sup>(15)</sup>.

After casting, the ring was left for bench cooling to room temperature, and then the sample were removed from the muffle, cleaned from the investment, sand blast with 250 µm aluminum oxide to remove any remaining investment and the oxide layer which covers the sample due to casting. Fig. (3.18), then the sprues would be sectioned by the carbondum disc mounted on laboratory hand piece. Each individual sample was checked, for incomplete casting, any nodules and any surface irregularity, sample with major defects were discarded and replaced with new one, minor defect such as small nodules at the edge of the disc, were removed by stone bur.

The samples were finished to their final dimensions and any irregularities after casting were removed from the cast specimen with stone bur and they were gradually polished on the flat surfaces with silicon carbide paper (600 grit) manually for 50 second at 10 cm distance in straight line on a flat table.<sup>(16)</sup>

All the finishing steps were done under running water to overcome heat generation, then evaluation of the surface polishing was performed by making profilometric records with the surface roughness instrument fig (7).

Profilometric parameter (Ra) was selected for this study samples, which gave a roughness between (0.09-0.13

µm) were used and any sample with higher roughness was re-polished and their roughness was reevaluated again. The samples thickness was calibrated with a metal caliper device (8mm×4.5mm).

### Metal Surface Treatments of the Tested Group

**Group A and Group A<sub>t</sub>:** The samples of these groups received no treatment and serve as control groups.

**Group B and B<sub>t</sub>:** The samples were treated with a chemical bonding agent (metal primer II); the metal primer II, as single liquid, was directly applied to the alloy surface with a disposable brush for (15 second) and then air dried for (5 second) before added the opaque<sup>(17)</sup>. Fig. (8)

**Group C and C<sub>t</sub>:** Each sample in these groups were individually micro – abraded, air abrasive with 50 µm aluminum oxide abrasive at air pressure of (60 psi) for (15 second) at a distance (10 mm). (18).

The sample was fixed in especially designed fixture (a holder) placed inside the sandblast machine at (10mm) distance away from the nozzle, the nozzle was kept perpendicular to the metal surface (ruler was to standardize the distance between the nozzle and the sample). Fig.(3.23). The samples then placed in distilled water in an ultrasonic cleaner for 10 minutes and then dried, stored dry until they were bonded<sup>(20,21)</sup>.

**Group D and D<sub>t</sub>:** Specimens of these groups were first air abraded similar to group (C,C<sub>t</sub>) samples, cleaned with distilled water in an ultrasonic cleaner for (10 minutes), then in these groups the samples treated with chemical bonding agent (metal primer II) that was

applied to the metal surface before added the opaque for each sample, the metal primer II was applied by disposable brush for 15 second and then air dried for (5 second).

**Group E, E<sub>t</sub>:** In this group the samples were treated as in group (C, C<sub>t</sub>) but used 110 µm aluminum oxide abrasive.

**Group F, F<sub>t</sub>:** Specimens of these group were first air abrade similar to group (E, E<sub>t</sub>) then the samples were treated with metal primer II as in group (B, B<sub>t</sub>).

**Group G and G<sub>t</sub>:** The samples were treated as in group (C, C<sub>t</sub>) but used 250µm aluminum oxide abrasive.

**Group H and H<sub>t</sub>:** Specimen of these groups was first air-abraded similar to group (G, G<sub>t</sub>) then metal primer II were applied in the same manner as in group (B,B<sub>t</sub>).

### Acrylic packing

The surface of the metal to be veneered was coated with opaque (liquid–powder mixing ratio about 2:1 by volume). Thin layer of opaque were applied, let it to dry for approximately (15 minutes) before added body acrylic facing according to manufactures instruction. The thickness of the opaque layer was measured with metal caliper device and scored (0.5mm)<sup>(12)</sup>. Fig.(9,A). To correct the pressure, size and shape of acrylic veneer used copper ring (8mm in diameter x2mm in high). The inner surface of the ring coated or painted by separating medium and correct with sample by used stick tap with specimens before added the dentin<sup>(12,13)</sup>. Fig. (9, B).

The mixing of the dentin was done in the mixing jars for a sample 10 to 15 drops of liquid were put into the mixing jar and dentin powder was added until all the liquid has been absorbed (liquid –powder mixing ratio approximately 1:3 by volume). Stir briefly and vigorously an agate or glass

spatula (the mixing time about 1 minute). Close Jar and allow to ripen. The dough is ready for use when it no longer sticks to the spatula. Applied dentin to the whole object to be veneered, which has been covered with opaque, and adapted the dentin with spatula moistened with liquid. Mix incisal dough. Mixing ratio approximately 1:2 by volume (i.e thinner than for dentin), stir dough thoroughly, and do not allow to ripen (in contrast to dentine), apply with spatula and spread. The total thickness of acrylic about (8mm in diameter and 2mm in high) <sup>(12)</sup>.

After modeling of acrylic facing was completed, the polymerization or curing the acrylic was done in the ivomat which was filled by water and then cure the acrylic facing for (10min.) at (120°C), (6 bar) pressure (according to manufacture's instruction). In the first removed the copper ring from the sample, then the acrylic was finished by carbide bur to remove the excessive amount of acrylic then stone bur used to standardize the thickness of the facing followed by sand paper used for smoothing. The final thickness of acrylic were checked by a vernier, The polishing was done with bristle brush and wet pumice using dental lath polishing machine at speed (30.000 rpm) finally used universal polishing past with cotton buff brush mounted on the polishing machine at the same speed. The final shape of each group as show in Fig. (10).

### B-Thermo cycling

To simulate oral condition, many of the test samples are subjected to numerous temperature cycle in water <sup>(21)</sup>.

The samples in the second category with thermocycling (A<sub>t</sub>, B<sub>t</sub>, C<sub>t</sub>, D<sub>t</sub>, E<sub>t</sub>, F<sub>t</sub>, G<sub>t</sub> and H<sub>t</sub> groups) the samples were stored inside incubator at room temperature water (37°C) for (24

hours) then they were placed alternatively in water at (5°C-55°C±1) for 60 second (30 second at each temperature) in a thermo cycling machine. The thermo cycling was repeated (1000 time) before subjecting the samples to the testing machine <sup>(22,23)</sup>.

### C-Shear Bond Strength Testing

Shear bond strengths were tested with zwick - instron universal testing machine, using the chisel shaped rod with a cross head speed of 0.5mm/min <sup>(6)</sup>.

The tested specimens were placed in the lower member (Jaw) of the testing machine so that the chisel rod was positioned at 90° at the interface between the edge of the metal and the resin. The specimens secured tightly in place to ensure that the specimens were always be kept at 90° in the vertical plan. <sup>(24)</sup>.

#### bond strength test.

The specimens were then stressed to failure. The force of bond failure was recorded in Newton which has been divided by the surface of bonded area to obtain the shear bonded strength calculated in MPa <sup>(10,21)</sup>.

$$SBS = F/SA = MPa$$

SBS = shear bond strength

F = force (N)

SA = surface area of bonded site (mm<sup>2</sup>).

The surface area of bonded site was calculated as follows:

$$SA = R^2 \times 22/7 = mm^2 \quad (\text{where } R^2 = \text{square radius}).$$

$$SA = 16 \times 22/7 = 50.28 mm^2$$

The shear bond strength data obtained were recorded and submitted to statistical analysis.

The mode of failure of each sample was evaluated, two methods were followed:

- 1 - Visual examination (unaided eye) by two persons in order to establish

the percentage of residual veneer on the metal.

- 2 - Under traveling light microscope (X4 magnification) <sup>(25)</sup>

## D-Statistical Analysis

The statistical methods were used in order to analyze and assess the result, included:

### I - Descriptive Statistics

- 1 - Statistical tables
- 2 - Mean of value
- 3 -Standard deviation
- 4 -Standard error for mean value (SD)
- 5 -Minimum and maximum reading
- 6 -Graphical presentation by (Bar - charts).

### II- Inferential Statistics

These were used in order to accept or reject the statistical hypotheses, they include:

- 1 -Student test (t-test) for comparison between two groups.
- 2 -Least significant difference (LSD) for multiple comparisons after (ANOVA) test.

## Results

### Shear Bond Strength Test Results

Results of shear bond strength calculated in (Mpa) were obtained for sixty four specimens in the first category (without thermocycling), which include the eight groups of the metal surface treatment that were tested in dry conditions.

The results of shear bond strength calculated in (Mpa) were obtained for the sixty four specimens in the second category (with thermocycling), which include the eight groups of the metal surface treatments that were tested in wet condition

The mean SBS, standard deviation, standard error, and maximum and

minimum values for each group (without TC) are listed in Table (1).

Inferential statistical methods represented by analysis of variance "ANOVA" test show that there are statistically highly significant differences at  $P < 0.01$  (was recorded between at least two different groups, Table (2).

The source of difference is investigated by further complement analysis of data by using LSD (Least Significant Difference) test to examine the differences between the different pairs of the eight groups as shown in Table (3).

The mean SBS, standard deviation, standard error, and maximum and minimum values for each group (with TC) are listed in Table (4).

Inferential statistical methods represented by analysis of variance "ANOVA" test show that there are statistically highly significant difference at  $P < 0.01$  was recorded between at least two different groups, Table (5)

The source of difference is investigated by further complement analysis of data by using LSD (Least Significant Difference) test to examine the differences between the different pairs of the eight groups as shown in Table (6).

Student test (t-test), was performed to examine the difference between each group with and without thermocycling ((A, A<sub>t</sub>), (B, B<sub>t</sub>), (C, C<sub>t</sub>), (D, D<sub>t</sub>), (E, E<sub>t</sub>), (F, F<sub>t</sub>), (G, G<sub>t</sub>) and (H, H<sub>t</sub>)) as shown in Table (7)

### Mode of Failure

After completion shear bond strength testing procedure all the specimens (with and without thermocycling) were examined visually and under a reflected light microscope (X4 magnification) in an effort to determine the type of bonding failure.

Three types of bonding failure were presented at the fracture site:

- Adhesive failure: Failure occurred at the interface between acrylic resin and metal. Fig. (15).
- Cohesive failure: Failure occurred in the bulk of the acrylic resin. Fig. (16).
- Cohesive / adhesive failure (mixed failure): A combination of both (adhesive and cohesive failure) Fig. (17).

Table (8) the percentage of mode of failure of each group of shear bond strength as influenced by different surface treatments.

## Discussion

*In group A* the control group showed low bond strength values relative to other group as shown in table (1). This is due to the fact that resinous materials for the veneer of fixed prostheses commonly require mechanical retention on the metal substructure because there is no chemical adhesion, and the potential for separation of the resin from the metal is also influence by the difference in thermal expansion coefficients of the resin and metal (11,13).

This study agrees with (2), who said that "on polished specimens, no bonding was achieved, because silicon oxide particles were not embedded in the surface of the alloy.

Also agree with (26), they found that the low shear bond strength values in polishing group when used the resin with two types of alloy (Ni-Cr and Ag-Pd) comparison with other group treated by 37 $\mu$ m Al<sub>2</sub>O<sub>3</sub> and 250 $\mu$ m Al<sub>2</sub>O<sub>3</sub> air abrasion .

This was also supported by (27), they found that the specimens of the polish group presented the

lowest SBS values compared with other group (polished with primer ,sandblasted without primer ,and sandblasted with primer)between the rely x –cement and polished surface of the Co- Cr –Mo alloy .

On the other hand the results obtained in present study disagree with those obtained by (20), he found that the no surface treatment showed the best treatment for Cpti were bonded with chemically cured resin comparison with other treatment (air abrasion with aluminum oxide, acid etch by mixture of 50% HNO<sub>3</sub> and 50% HCL and combination of air abrasion +acid etching.

*In group B* (polished with metal primer II )the results showed that the chemical bonding agent (metal primer II)shows no improvement in the SBS at (p value >0.05 )compared with the control group .And it gave the lowest mean SBS value compared with all other groups shown in table (1) &(4).

The results of the present study was in agreement with that obtained by (28), where they found that different chemical bonding agent in general gave low SBS .

This was also supported by (13), he found that the chemical bonding agent alone will not improve the bond strength and it requires a combination of other retentive system to ensure good and stable bond.

This study agree with (6), they found that the bond strength of resin to type IV gold alloy specimens polished with 600Sic paper could not be improved even through the silica system.

Also agree with (29), they found that bond with acrylic veneered materials were provide with only mechanical retention (bead and air abrasion).



Also <sup>(30)</sup>, evaluated the effect of adhesive on the bond strength of a porcelain veneer to base metal alloy; they found that untreated samples exhibited higher shear bond strength value than other adhesively treated groups.

The results of the present study disagree with <sup>(13)</sup>, they studied the effect of metal primers on adhesive bonding with gold alloys, they found that this primer improved the SBS and they explained the results that these primers react well with the precious alloy used in their study.

The results of the present study disagree with results obtained by <sup>(6)</sup>, who reported that the bond strength of resin to Cpti alloy treated with 600grit sic and siloc system showed higher shear bond strength compared with 50 $\mu$ mAl<sub>2</sub>O<sub>3</sub> without the siloc system.

The results also disagree with the results obtained by <sup>(9,17)</sup> they found that specimens treated with different chemical bonding agents had significantly higher bond strength than non treated group (control group), this may be explained by that they used different types of adhesive agents other than the metal primer II, or because they have used these agents to bond Co-Cr alloy to cold cure acrylic, while in the present study heat cured acrylic was used and so due to the different materials used different results was obtained.

The results also showed a highly significant difference at (P<0.01) between metal primer II group and air abrasion groups as show in table (1) and this may be explained by that the air abrasion produced micromechanical retention areas on the metal surface, such areas allows

deeper penetration of the acrylic resin into the metal surface and hence increase the bond strength at the interface such retentive areas are not present with metal primer (chemical bonding agent).

One of the easy methods of enhancing bond strength is roughening the surface by air abrasion.

*In group C, E, and G*, the results showed that air abrasion raised the mean bond strength values higher than that of the control group and metal primer II group (chemical bonding agent) as show in table (1) & (4).

This improvement in the SBS may be due to the fact that the process of air abrasion cleaning the alloy surface area, remove unfavorable contamination, produced suitable and uniform micro retention.

Each of these factors has been shown to increase the wettability of the metal surface and bonding surface area <sup>(26,32)</sup>

The results are supported by <sup>(33)</sup>, who evaluated the effect of surface roughness on retention of resin – metal casting interface, and found that air abrasion provided the surface roughness topography with greatest resistance to shear stress.

The results also agree with those obtained by **(11)**, who found that air abrasion increased surface area, expanded the energy, and also heightened activity of the alloy surface area, thus results in increased bond strength.

These results are in agreement with **(26)**, who found that the surface treatment of the metal by sandblasting with Al<sub>2</sub> O<sub>3</sub> particles (37-250  $\mu$ m) has improved the effectiveness of the surface area of the metal and increased the resin – metal bond strength. The resin

alloy bond strengths improved remarkably after sandblasting.

Similar results obtained by (7), when they evaluated the effectiveness of the metal surface treatment in controlling microleakage of the acrylic resin – metal interface, and found significant reduction in microleakage, which means significant improvement of the bond strength.

This result was in agreement with that obtained by (34), who reported that  $Al_2O_3$  increased the reaction on the metal surface with the resin and created micromechanical retention that increased the bond strength.

Previous studies reported that the alumina not only affected the micromechanical roughness of the surface but it left alumina particle embedded in the surface and ultrasonic cleaning result in only minor removal of the embedded alumina, which effect on bond strength.

The results of the present study are not consistent with the results obtained by (12), who said that "air abrasion of the metal by it self is not recommended as a retentive system because of retention inconsistency.

Also this study disagree with (20), he found that the air abrasion gave the low shear bond strength than no surface treatment for Cpti were bonded with chemically cured resin.

In comparison between the effect of air abrasion with different grain size (C, E, and G) groups, the results showed that the better SBS obtained by 50  $\mu m$  followed by 110  $\mu m$  and finally by 250 $\mu m$  this is may be due to that air abrasion with 50 $\mu m$   $Al_2O_3$  provided a metal resin bonded with lower resin film thickness and (35), mentioned that

when a thin film is created between two materials, that makes the system stronger than bulk properties of adhesive. Or may be due to the surface treated with 50 $\mu m$  aluminum oxide less effected of ultrasonic cleaned and surface containing a significant amount of  $Al_2O_3$ , which effects bond strength and gave best bond.

This agree with (9) they concluded that sandblasting the base alloy surface that resulted in the surface containing a significant amount of aluminum oxide particles, which effect bond strength has been reported. Or may be due to the configuration of roughness and under cut created by 50 $\mu m$  more stable for retention than other size.

But this results disagree with (6), they found that greatest shear bond strength between resin and Cpti titanium when treated by 250 $\mu m$   $Al_2O_3$ . Also they found no difference in shear bond strength between resin and type IV gold alloy when treated by 50 $\mu m$  and 250 $\mu m$   $Al_2O_3$ .

**In group (D, F, and H)** the results of (air abrasion plus chemical bonding agent) showed a highly significant difference at (p value <0.01) with control and metal primer II group (A, B) group as shown in table (1).

Although the (air abrasion plus metal primer II) effect on the SBS mean value was more than air abrasion effect alone as shown in table (1), & (4).

The results indicates that (micromechanical /chemical pretreatment of the metal surface is required to produce stronger bonds than those achieved by each treatment alone.

The results in the present study agreement with (17), they reported that aluminum oxide embedded on

the surface of the alloy after air abrasion embedded aluminum oxide, which can create chemical bonds with the chemical adhesive agent and enhance the bond strength of the metal-resin interface.

This result also supported by <sup>(9)</sup>, they reported that the application of any one of the primer significantly improved the shear bond strength of the resin to any of the cast metal tested (Cp titanium, Ti-6Al-4V, and Co-Cr alloy).

Also agree with <sup>(27)</sup>, they concluded that the best outcomes of retention were found for the sandblasted with primer. Alloy primer application was effective in the formation of chemical bonding between the tested resin and the cobalt-chromium alloy; However, this bond strength did not overcome the force of the micromechanical union reached by sandblasting with aluminum oxide.

In comparison between all groups the results showed the best shear bond strength obtained by 50 $\mu$ m aluminum oxide plus metal primer II. This is due to the fact that the resinous materials for the veneers of fixed prostheses commonly require mechanical retention on metal substructure, and in the original the 50 $\mu$ m of Al<sub>2</sub>O<sub>3</sub> gave the higher SBS compared with other particles (110 $\mu$ m and 250  $\mu$ m Al<sub>2</sub>O<sub>3</sub>).

#### **The Effect of Thermocycling :**

After thermocycling, the results showed that the group D<sub>t</sub>, F<sub>t</sub>, and H<sub>t</sub> (air abrasion plus chemical bonding agent group) are more stable and durable SBS compared with control group and metal primer II group (A<sub>t</sub>, B<sub>t</sub>) as shown in table (4).

This improvement in SBS may be explained as the sandblasted with

primer improved the SBS between the resin and alloy before and after thermocycling.

Also in the group C<sub>t</sub>, E<sub>t</sub>, and G<sub>t</sub> (air abrasion) showed a more stable and durable SBS compared with control group as shown in table (4). This improvement in SBS with TC may be explained as the microretention area created by air abrasion allows deeper penetration of acrylic into the micromechanical irregularities so that providing more resistance to TC than the control group and metal primer II group (A<sub>t</sub>, B<sub>t</sub>).

In group B<sub>t</sub> (metal primer II), the metal primer II showed no improvement in the SBS compared with the control group as shown in table (4).

In table (6) in comparison between the same group before and after thermocycling, the results show a highly significant difference at (p<0.001) present between the control groups (A, A<sub>t</sub>). But the results show a significant difference at (p value < 0.05) present in group (B, B<sub>t</sub>) and (C, C<sub>t</sub>) (E, E<sub>t</sub>) and (G, G<sub>t</sub>).

While in group air abrasion plus metal primer II (D, D<sub>t</sub>), (F, F<sub>t</sub>), and (H, H<sub>t</sub>) group shows non-significant difference at (p value > 0.05).

This result explained that the more stable and durability of SBS shown in group air abrasion plus metal primer II.

This was supported by <sup>(28)</sup>; they found that chemical bonding minimized gap formation at the metal-resin interface.

This study also agrees with <sup>(17)</sup>, they found all the primers examined improved the shear bond strength between the resin and alloy compared with non-primed specimens before and after thermocycling. However, they reported that the bond strengths of resin to alloy primed with Cop (copied opaque primer) or MPII (metal primer II) primers were significantly greater

than those of specimens primed with AB(Acryl bond) or MR(MR bond) primers and non primed controls .

Also agree with <sup>(36)</sup>, they concluded that the combined use of the resin cement and an appropriate adhesive primer spare the use of complicated surface treatments during cementation of adhesive bridge ,which minimize the formation of marginal gaps and increase the clinical durability of restoration .

Also <sup>(13)</sup> they demonstrated that thermocycling caused a significant drop of shear bond strength. They also showed that thermocycling speed up the diffusion of water in between resin and metal .Changing the temperature creates stress at the interface of the two materials because of the different coefficient of thermal expansion ,which results in marginal gaps and detachment ;this may be related to the more intensive thermocycling and increased number of cycles performed over a lesser time period .

These study also disagreement with <sup>(10,26)</sup> they found that thermocycling caused a significant decrease in shear bond strength of metal –resin bonds for most of the tested.

### Mode of Failure

In table (7) ,the results shows that the untreated group and metal primer II group (without and with thermocycling ) , the bond failed mostly adhesively, since these groups revealed low SBS values compared with air abrasion group and air abrasion plus chemical bonding agent (metal primer II) group. This happened because of the fact that bonding between resin and metal did not overcome the cohesive resistance of the resin and the bond failed at the interface of the resin and metal. This results agreed with <sup>(9,27)</sup>.The results showed in air abrasion group with different grain size ( $50\mu\text{mAl}_2\text{O}_3$

, $110\mu\text{m Al}_2\text{O}_3$ ,  $250\mu\text{m Al}_2\text{O}_3$  ) that the bond mostly cohesively or /mixed ,since this groups revealed high SBS value compared with untreated group and metal primer II group.The results also showed that for the air abrasion plus metal primer II group (without and with TC ) the bond failed cohesively for nearly all the specimens ,since this group showed the highest SBS values among other groups this indicated that bonding occurred between the acrylic resin and the metal and that the failure occurred in the acrylic resin rather than the interface of the acrylic and metal .These results agree with <sup>(10,13)</sup>.

### Conclusions

On the basis of the results arrived at, the following conclusions can be drawn:

- 1 - Statistically significant difference in mean shear bond strength was observed of different groups with different grain size of aluminum oxide and the best outcomes of retention were found for the sandblasted treated with  $50\mu\text{mAl}_2\text{O}_3$ .
- 2 - The use of mechanical /chemical retention system results in a greater bond even with thermocycling, than only mechanical retention systems, especially  $50\mu\text{m Al}_2\text{O}_3$  plus chemical bonding agent enhanced the shear bond strength of metal-resin interface.
- 3 - The effect of chemical bonding agent (metal primer II ) on the shear bond strength values is negligible compared with untreated group and it require air abrasion of the metal surface to act in a perfect form.
- 4 - The thermocycling caused a highly significant drop in shear bond strength of untreated specimens and caused a significant drop in shear

bond strength of metal primer II group and air abrasion group but the most durable bond strength to Ni- Cr alloy obtained with air abrasion plus metal primer II.

- 5 - In general ,the mode of failure are mostly adhesive in the untreated group and metal primer II group and are mostly cohesive in air abrasion group and air abrasion plus metal primer II .

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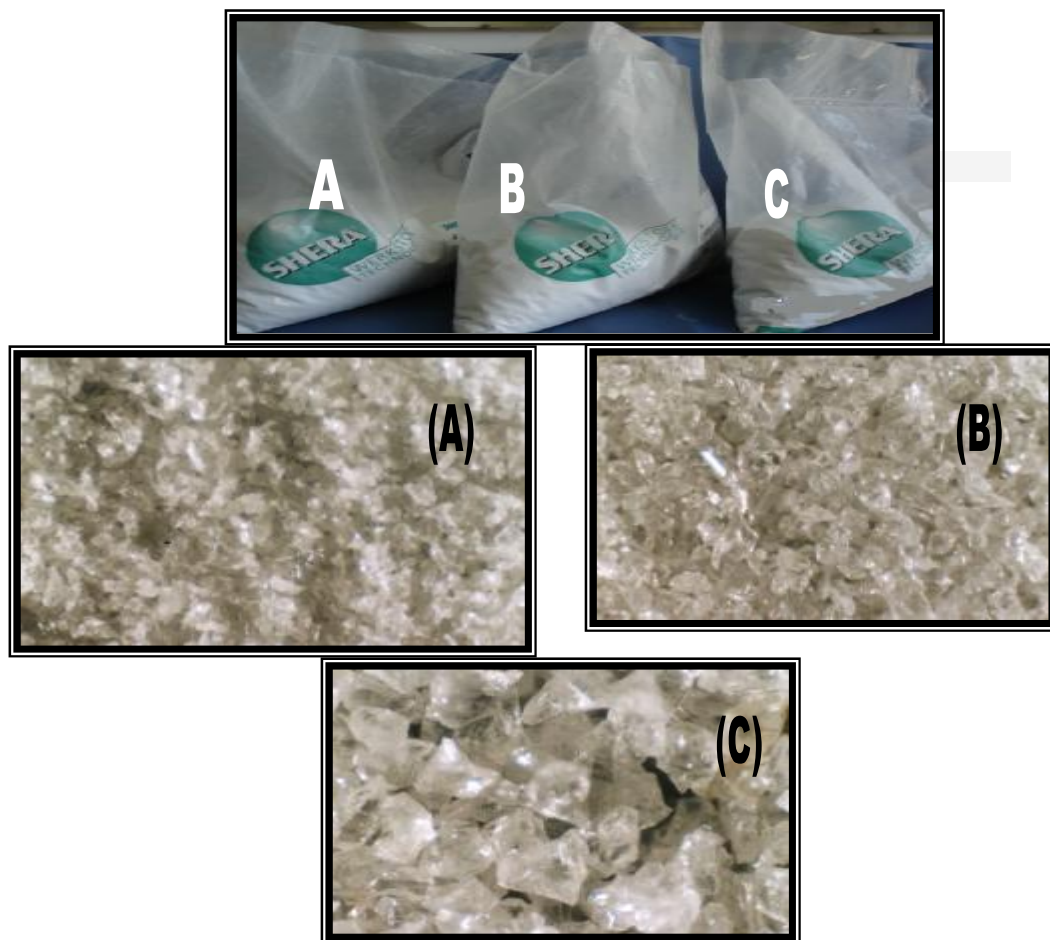


Fig. (1): Aluminum oxide powder (A)  $50\mu\text{m Al}_2\text{O}_3$  (B)  $110\ \mu\text{m Al}_2\text{O}_3$  (C)  $250\mu\text{m Al}_2\text{O}_3$ .

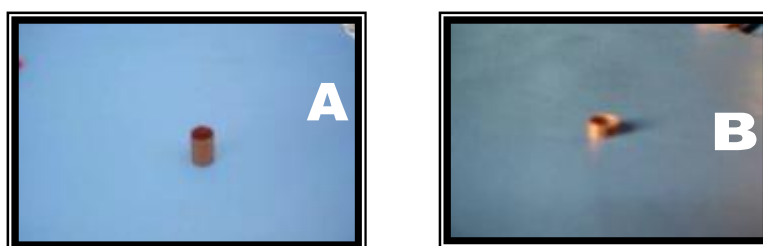


Fig. (2): Copper rings (A) ring for wax pattern preparation (B) ring for correct the dimension of the acrylic



Fig. (3): Profilometric device



Fig. (4): Thermocycling device



Fig. (5): Instron universal testing machine.



Fig. (6): Wax pattern preparation.



Fig. (7): Surface roughness tester (profilometer).



Fig. (8): Applying of the metal primer II.

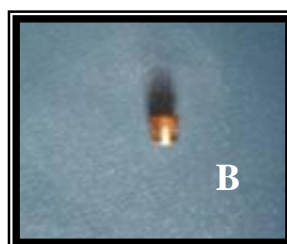


fig.(9):

(A)the sample with opaque layer

(B) Alloy sample with ring



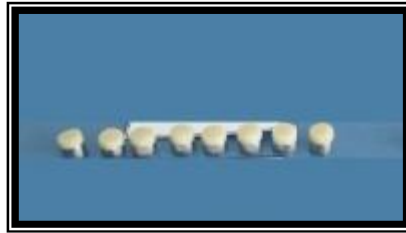


Fig. (10): The final shape of each group before test.

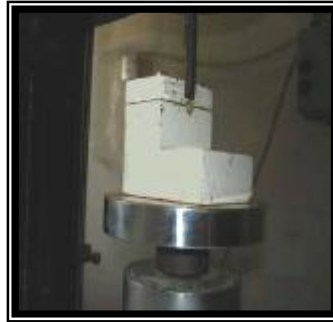


Fig. (11): Specimens under shear

Table (1) Descriptive statistic of different group (without TC) .

Studied groups	No.	Mean	Std. Deviation	Std. Error	Min.	Max.
Polishing (A)	8	1.557	0.238	0.084	1.17	1.95
Metal primerII(B)	8	1.825	0.230	0.081	1.46	2.14
50 $\mu$ m (C)	8	4.632	0.504	0.178	3.90	5.46
50 $\mu$ m +MP II(D)	8	5.312	0.584	0.206	4.19	5.95
110 $\mu$ m (E)	8	4.155	0.433	0.153	3.60	4.87
110 $\mu$ m +MPII(F)	8	4.763	0.466	0.164	3.99	5.26
250 $\mu$ m (G)	8	3.446	0.396	0.140	3.02	4.09
250 $\mu$ m+MPII(H)	8	4.177	0.322	0.114	3.80	4.68
Total	64					

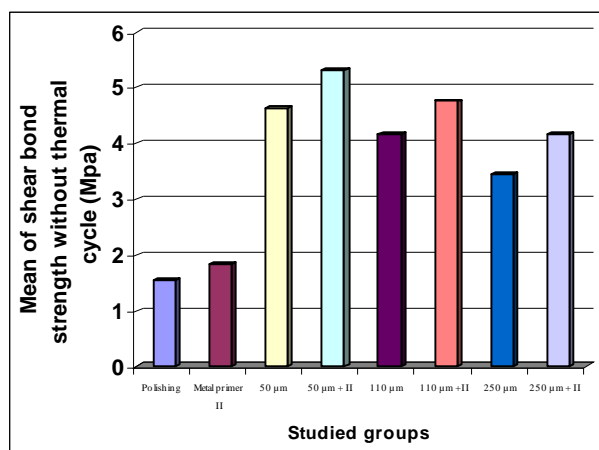


Fig. (12) Mean distribution of shear bond strength without thermal cycle \according to the studied groups.

Table (2) ANOVA test for metal surface treatment groups (without TC)

ANOVA test	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	105.850	7	15.083	78.962	0.00
Within Groups	9.602	56	0.171		Highly Sig. (P<0.01)
Total	115.182	63			

Table (3) Least significant difference (LSD) test for the shear bond strength using different metal surface treatments (without TC).

LSD (F – test)								
Studied groups	Polishing	Metal primerII	50 $\mu$ m	50 $\mu$ m + II	110 $\mu$ m	110 $\mu$ m +II	250 $\mu$ m	250 $\mu$ m + II
	(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)
Polishing (A)	-	NS	HS	HS	HS	HS	HS	HS
MetalprimerII(B)	-	-	HS	HS	HS	HS	HS	HS
50 $\mu$ m (C)	-	-	-	HS	S	NS	HS	S
50 $\mu$ m +MP II(D)	-	-	-	-	HS	HS	HS	HS
110 $\mu$ m (E)	-	-	-	-	-	HS	HS	NS
110 $\mu$ m+MPII(F)	-	-	-	-	-	-	HS	HS
250 $\mu$ m (G)	-	-	-	-	-	-	-	HS
250 $\mu$ m+MPII(H)	-	-	-	-	-	-	-	-

NS = Non significant difference (P>0.05) S = Significant difference (P<0.05)

HS = Highly significant difference (P<0.01)

Table (4) Descriptive statistic of different group (with TC).

Studied groups	No.	Mean	Std. Deviation	Std. Error	Min.	Max.
Polishing (A <sub>i</sub> )	8	1.191	0.214	0.075	0.97	1.56
Metal primerII(B <sub>i</sub> )	8	1.515	0.245	0.086	1.26	1.95
50 $\mu$ m (C <sub>i</sub> )	8	4.128	0.284	0.100	3.70	4.48
50 $\mu$ m+MP II (D <sub>i</sub> )	8	5.067	0.568	0.200	3.99	5.75
110 $\mu$ m (E <sub>i</sub> )	8	3.652	0.368	0.130	3.12	4.09
110 $\mu$ m+MPII (F <sub>i</sub> )	8	4.360	0.521	0.184	3.60	4.97
250 $\mu$ m (G <sub>i</sub> )	8	2.742	0.703	0.248	1.75	3.80
250 $\mu$ m+MPII (H <sub>i</sub> )	8	3.717	0.522	0.184	3.21	4.58
Total	64					

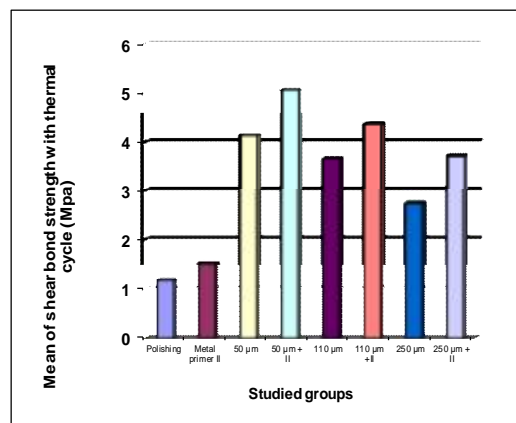


Fig. (13): Mean distribution of shear bond strength with thermal cycle according to the studied groups.

Table (5) ANOVA test for metal surface treatment groups (with TC).

ANOVA test	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	105.415	7	15.059	71.504	0.00
Within Groups	11.794	56	0.211		Highly Sig. (P<0.01)
Total	121.665	63			

Table (6) The result of multiple comparison test (LSD) of shear bond strength with thermal cycle according to the studied groups.

LSD (F-test)								
Studied groups	Polishing (A <sub>t</sub> )	Metal Primer II (B <sub>t</sub> )	50 µm (C <sub>t</sub> )	50 µm + II (D <sub>t</sub> )	110 µm (E <sub>t</sub> )	110 µm +II (F <sub>t</sub> )	250 µm (G <sub>t</sub> )	250 µm + II (H <sub>t</sub> )
Polishing (A <sub>t</sub> )	-	NS	HS	HS	HS	HS	HS	HS
MetalprimerII (B <sub>t</sub> )	-	-	HS	HS	HS	HS	HS	HS
50 µm (C <sub>t</sub> )	-	-	-	HS	S	NS	HS	NS
50 µm +MP II (D <sub>t</sub> )	-	-	-	-	HS	HS	HS	HS
110 µm (E <sub>t</sub> )	-	-	-	-	-	HS	HS	NS
110 µm +MPII(F <sub>t</sub> )	-	-	-	-	-	-	HS	HS
250 µm (G <sub>t</sub> )								HS
250 µm +MPII(H <sub>t</sub> )	-	-	-	-	-	-	-	-

Table (7) Comparison study between with &amp; without thermal cycle in every one of studied groups by shear bond strength.

Studied groups	N	Mean	Std. Deviation	Std. Error	Mini.	Maxi.	C.S by (t-test)
(A)	8	1.557	0.238	0.084	1.17	1.95	0.006 HS
(A <sub>t</sub> )	8	1.191	0.214	0.075	0.97	1.56	
Total	16						
(B)	8	1.825	0.230	0.081	1.46	2.14	0.021 S
(B <sub>t</sub> )	8	1.515	0.245	0.086	1.26	1.95	
Total	16						
(C)	8	4.632	0.504	0.178	3.90	5.46	0.024 S
(C <sub>t</sub> )	8	4.128	0.284	0.100	3.70	4.48	
Total	16						
(D)	8	5.312	0.584	0.206	4.19	5.95	0.409 NS
(D <sub>t</sub> )	8	5.067	0.568	0.200	3.99	5.75	
Total	16						
(E)	8	4.155	0.433	0.153	3.60	4.87	0.025 S
(E <sub>t</sub> )	8	3.652	0.368	0.130	3.12	4.09	
Total	16						
(F)	8	4.763	0.466	0.164	3.99	5.26	0.125 NS
(F <sub>t</sub> )	8	4.360	0.521	0.184	3.60	4.97	
Total	16						
(G)	8	3.446	0.396	0.140	3.02	4.09	0.027 S
(G <sub>t</sub> )	8	2.742	0.703	0.248	1.75	3.80	
Total	16						
(H)	8	4.177	0.322	0.114	3.80	4.68	0.053 NS
(H <sub>t</sub> )	8	3.717	0.522	0.184	3.21	4.58	
Total	16						

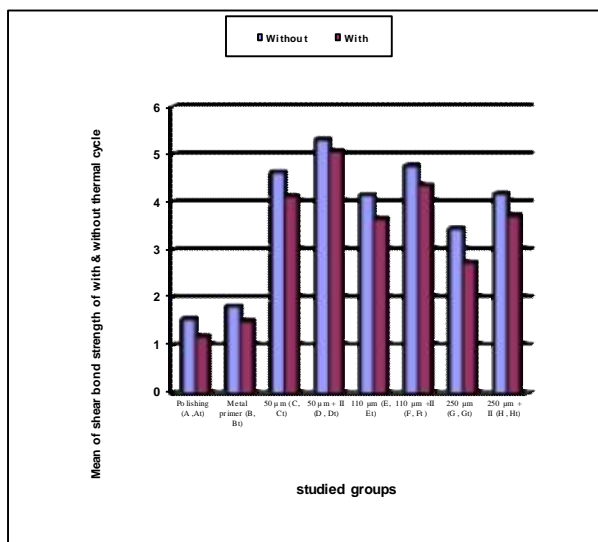


Fig. (14) Bar graph of mean SBS of different type of metal surface treatments with different storage categories (with & without thermocycling)



Fig. (15): Adhesive failure (X4)



Fig. (16): Cohesive failure (X4).



Fig. (17): Mixed failure (X4).

Table (8): The percentage of Mode of failure without and with thermocycling.

Type of thermal cycle	Type of surface treatment (groups)	Mode of failure %		
		Adhesive	Cohesive	Mixed
Without thermocycling	Untreated group (A)	100	0	0
	Metal primer II group (B)	87.5	0	12.5
	50 $\mu$ m Al <sub>2</sub> O <sub>3</sub> group (C)	0	62.5	37.5
	50 $\mu$ m Al <sub>2</sub> O <sub>3</sub> + metal primer II group (D)	0	75	25
	110 $\mu$ m Al <sub>2</sub> O <sub>3</sub> group (E)	12.5	50	37.5
	110 $\mu$ m Al <sub>2</sub> O <sub>3</sub> + metal primer II group (F)	0	75	25
	250 $\mu$ m Al <sub>2</sub> O <sub>3</sub> group (G)	25	37.5	37.5
	250 $\mu$ m Al <sub>2</sub> O <sub>3</sub> + metal primer II group (H)	0	62.5	37.5
With thermocycling	Untreated group (A <sub>t</sub> )	100	0	0
	Metal primer II group (B <sub>t</sub> )	100	0	0
	50 $\mu$ m Al <sub>2</sub> O <sub>3</sub> group (C <sub>t</sub> )	12.5	62.5	25
	50 $\mu$ m Al <sub>2</sub> O <sub>3</sub> + metal primer II group (D <sub>t</sub> )	0	75	25
	110 $\mu$ m Al <sub>2</sub> O <sub>3</sub> group (E <sub>t</sub> )	25	37.5	37.5
	110 $\mu$ m Al <sub>2</sub> O <sub>3</sub> + metal primer II group (F <sub>t</sub> )	0	62.5	37.5
	250 $\mu$ m Al <sub>2</sub> O <sub>3</sub> group (G <sub>t</sub> )	37.5	12.5	37.5
	250 $\mu$ m Al <sub>2</sub> O <sub>3</sub> + metal primer II group (H <sub>t</sub> )	25	37.5	37.5