

# **SHEAR BOND STRENGTH OF THREE ADHESIVE SYSTEMS TO AMALGAM, BASE METAL AND NOBLE METAL**

*AVALIAÇÃO DA RESISTÊNCIA DE UNIÃO DE TRÊS SISTEMAS ADESIVOS COM AMÁLGAMA, LIGA NOBRE E LIGA NÃO NOBRE*

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**T**his in vitro study evaluated the shear bond strength of three versatile bonding agents: Prisma Universal Bond3, All-Bond2 and Scotchbond Multi-Purpose bonded to three different metals: Dispersalloy, Litecast B and Lodestar. The metal surface treatment consisted of grinding with 600 grit silicon carbide paper and then air abrading with 50 micron aluminum oxide particles using 50 psi pressure for five seconds. All samples were stored in water at 37°C for at least 48 hours, then they were thermocycled between temperatures of 8°C and 48°C for a total of 2,500 cycles. Following thermocycling, the samples were stored in water for a 14-day period then subjected to shear testing. An Instron Machine was used, and testing was conducted with a cross head speed of 1.0 mm/min. The specimen were stressed to fracture. In order to assess mode of failure, the metal surfaces were examined under a light stereo microscope at a magnification of 40X.

Scotchbond exhibited the highest bond strengths to all metals. These strengths were significantly greater ( $p < 0.05$ ) than those of All-Bond2 and Prisma Universal Bond3. All Bond2 and Prisma Universal3 strengths were not significantly different ( $p = 0.05$ ). All agents exhibited the highest bond strengths to base metal. These strengths were significantly different ( $p < 0.05$ ) from bond strengths to amalgam and gold. Bond strengths to the latter metals were not significantly different ( $p = 0.05$ ). All samples presented adhesive failure at the metal/bonding agent interface.

**UNITERMS:** Bond strength; Adhesives; Metals.

## INTRODUCTION

Recently, the use of adhesive systems has become popular in bonding resins to metals. Achieving a good bond to a metal surface has always been difficult, and researchers are still trying to develop an adhesive that produces a reliable attachment to metals.

A number of new "multipurpose" bonding agents have recently been marketed. The manufacturers claim high bond strength to different kinds of metals and to tooth structure. However, little clinical research on longevity of the resin/metal bond has been described<sup>1</sup>.

It is generally accepted that no currently available product will produce a resin repair of a metal ceramic restoration that is equal in strength to an unbroken porcelain, with or without exposed metal, will frequently be the most practical procedure for many clinical situations.

Another clinical situation in which the ability to bond a composite resin to a metal substrate is desirable would be the improvement of the cosmetic appearance of visible amalgam restorations.

Three new bonding agents: All Bond2, Prisma Universal Bond3, and Scotchbond Multi-Purpose claim good bonding to diverse substrates such as dentin, enamel, resin, metal and porcelain. The purpose of this study was to evaluate the shear bond strength of these three adhesive systems to three metals: base metal, noble metal, and amalgam.

## MATERIALS AND METHODS

### *Preparation of Metal Substrate*

Ninety specimens were fabricated for this study: 30 specimens with base metal alloy, 30 specimens with noble metal alloy and 30 with amalgam alloy. Each group was divided into three sub-groups of 10 specimens and one of the three bonding agents was applied to each sub-group. The specimens were thermocycled and stored in water for 14 days prior to testing.

A metal mold was fabricated for condensation of an admixed amalgam alloy, Dispersalloy (The L.D. Caulk Co., Div. of Dentsply International Inc., Milford, Del.). Within the mold there was a tapered cavity measuring 7.5 mm in diameter at one end, 10 mm in diameter at the other end and 3 mm in height. The same mold was used to fabricate wax patterns for casting two different crown and bridge alloys: Litecast B (Williams Gold, Buffalo, NY), a nickel chromium beryllium base metal

alloy and Lodestar (Williams Dental Co., Inc. Amhest, NY), a gold palladium alloy.

Three different bonding agents, Scotchbond Multi-Purpose (3M Dental Products Division St. Paul, MN), All Bond 2 (Bisco, Inc., Itasca, IL), and Prisma Universal Bond 3 (The L.D. Caulk Co.) were used to bond a composite resin (from the same manufacturers) to the metal specimens.

A Kerr Automix Computerized Mixing System, Model No. 23425 (Romulus, MI 48174, USA) was used to triturate the amalgam. The trituration time and speed followed the manufacturer's recommendations (3700 RPM for 12 seconds). The amalgam specimens were hand condensed (by the same person) in the metal mold described above. The specimens were kept in air at 37°C until they were mounted in a plexiglas mold. The amalgam specimens were made seven days prior to bonding with resin.

**Note:** Whenever the term "metal substrate" is used it applies to all three alloys in the study.

All metal substrates were mounted in the center of a plexiglas cylinder mold (19mm in diameter and 24 mm in length) with autopolymerizing acrylic resin. The 7.5 mm diameter end of each specimen was mounted toward the outside of the plexiglas cylinder mold. The specimen design was tapered so that they would offer more resistance to displacement from the acrylic during testing. After being mounted in the plexiglas cylinder molds, the specimens were stored in water.

The surface of all metal substrates was subjected to the following treatment: The exposed metal was ground with 600 grit silicon carbide paper under water using a Leco DS-20 (Leco Corp., St. Joseph, MI). The surface was then washed for 30 seconds and dried for 30 seconds. Then the surface of each specimen was sandblasted with an Airbrasive Unit (Model F, S.S. White Company, New York, N.Y.) utilizing 50 micron aluminum oxide particles with a pressure of 50 psi for five seconds. The tip of the air abrader was held at a distance of 20 mm above the metal substrate. The specimens were washed and dried with oil-free air and the bonding agents were applied following manufacturer's instructions.

### *Application of All-Bond 2 System to Metal Substrates*

The surface of the metal substrate was washed for 30 seconds and dried for 30 seconds. Primer A & B were mixed, and two coats of the mixture were applied to metal. The entire surface was dried for 5-6 seconds. A thin layer of dentin/enamel bonding resin was

applied and light cured for 20 seconds with a visible light (Coe-Lite, Imperial Chemical Industries PLC, Pharmaceutical Div., Macclesfield, Cheshire, England).

Bis Fil (Bisco) composite resin shade L was placed utilizing a split teflon mold (5 mm x 6 mm) and a silicone gasket with a 5 mm diameter hole in the center and an acrylic aligning device. The acrylic aligning device is large enough to accommodate the split teflon mold, the silicone gasket and the plexiglas cylinder mold inside. There are two screws on the side of the aligning device that are tightened to prevent the split teflon mold, the silicone gasket and the plexiglas cylinder mold from separating during the application of the resin to the metal. The plexiglas cylinder mold, the split teflon mold and the silicone gasket were oriented parallel to the acrylic aligning device's long axis so that bonding surface of the metal substrate was perpendicular to it. The silicone gasket was custom made in order to limit the spread of the bonding agent's components beyond the area designated for bonding. This was fabricated by first making a composite sample using only the split teflon mold. The junction of the composite and the bonding substrate was cleaned and any excess bonding resin was removed using a scalpel blade under 10 X magnification. Once this specimen was refined, a thin layer of poly (vinyl siloxane) impression material was carefully syringed around the junction of the composite specimen and the substrate, then the split teflon mold was pressed into place around the composite and toward the substrate. This layer of poly (vinyl siloxane) was placed against the bonding substrate under the split teflon mold and held there under slight compression prior to and during the placement of all bonding agents, and as a result, it served as a gasket, which confined the bonding agent components to the area selected for bonding. The resin was placed in 2 mm increments and each increment was cured for 40 seconds. The split mold was removed five minutes after curing and the samples were stored in water at 37°C.

#### **Application of Scotchbond Multi-Purpose System (SBMP) to Metal Substrates**

The surface of the metal substrate was washed for 30 seconds, and dried for 30 seconds. SBMP Etchant was applied for 15 seconds, rinsed for 40 seconds, then dried for 30 seconds. Litecast B and Loadstar specimens received a three coat application of Scotch Prime Ceramic Primer to the prepared surface, drying briefly between coats. Dispersalloy specimens received SBMP Primer application to the prepared metal surface. A thin layer of SBMP Adhesive was applied to all specimens

and light cured for 10 seconds. Composite resin, Z100 (shade A2), was placed as previously described for Bis Fil.

#### **Application of Prisma Universal Bond 3 System (PUB3) to Metal Substrates**

The surface of the metal substrate was washed for 30 seconds, and dried for 30 seconds. PUB3 primer was applied for 30 seconds and air dried for five seconds. PUB3 adhesive was applied on the prepared metal, thinned with air, and then light cured for 10 seconds. Composite resin, Prisma TPH (shade A2) was applied to the metal as previously described for Bis Fil.

#### **Storage and Thermocycling**

All the samples were stored in water at 37°C for at least 48 hours. They were thermocycled between temperatures of 8°C to 48°C for a total of 2,500 cycles with a dwell time of 30 seconds. Following thermocycling, the samples were stored in water at 37°C for a 14 day-period and then subjected to shear testing.

#### **Shear Bond Strength Test**

The shear bond strength Test employed has been used by the American Dental Association for adhesion testing as reported by Stanford et al.<sup>3</sup>

The test was conducted with a cross head speed of 1.0 mm/min. The specimens were stressed to fracture.

#### **Microscopy**

In order to assess mode of failure, the metal surfaces were examined under a light stereo microscope at a magnification of X40 (American Optical Binocular Microscope). Mode of failure was determined by estimating the amount of substrate free of restorative material (adhesive failure at the metal-bonding agent interface) as compared to the amount of material remaining on the metal surface (cohesive failure of this material). The test area was visually divided into eight segments and the percentages of adhesive and cohesive failure were estimated.

The failure mode of each specimen was recorded as follows:

A = Adhesive failure:  $\leq 75$  percent of the test area free of material

A-C = Adhesive - cohesive:  $>25$  to  $<75$  percent of the test area free of material

C = Cohesive failure:  $\leq 25$  percent of the test area free of material

R = Failure between resin and bonding agent

#### **Statistical Evaluation**

Bond strength measurements for all data were compared by a two-way analysis of variance (ANOVA) comparing the effects of using different metals and different bonding agents on the bond strength. A Neuman-

Keuls-Multiple Comparison Test was run to compare the bond strength among the groups. Mode of failure was expressed in percentage.

## RESULTS

A review of the original data showed that all groups, with exception of Scotchbond/amalgam, Scotchbond/base metal, and All-Bond/base metal had coefficients of variation less than 20 percent. These three groups had variations in excess of 30 percent.

Ten additional samples were made and tested for Scotchbond/amalgam, Scotchbond/base metal, and All-Bond/base metal. In each case, the coefficient of variation of the new data was lower than the original data.

T-Test of the original and new data indicated that the means were not significantly different. In the new data, the new groups had smaller standard deviation. Bartlett's Test indicated that the variances for the Scotchbond/amalgam groups were homogeneous and the new data was combined with the original data resulting in a sample size of  $n = 20$ . In the case of Scotchbond/base metal, and All-Bond/base metal the variances were not homogeneous and the original data was replaced with the new measurements.

The results from the bond strength tests are shown in table I and plotted graphically in figure 1. Highest bond strength were found for Scotchbond with all metals (10.51 - 13.37 MPa), and lowest bond strength were found with All-Bond when bonded to amalgam and gold (6.38 - 6.78 MPa).

The data were subjected to a Two-Way ANOVA. Both metal and bonding agent factors were significant at  $p < 0.001$ . The interaction term (bond agent/metal) was not significant  $p = 0.05$ .

Newman-Keuls Multiple Comparison was made on the mean values for the bond agents and the metals (table II). Scotchbond exhibited the highest bond strength, which was significantly greater ( $p < 0.05$ ) than All-Bond and Prisma Universal Bond 3 which were not significantly different ( $p = 0.05$ ). In the case of the metals, the base

metal exhibited the highest bond strength and was significantly different ( $p < 0.05$ ) than amalgam and gold, which were not significantly different ( $p = 0.05$ ).

All the specimens tested presented adhesive failure between the metal and bonding agent interface.

## DISCUSSION

Three bonding agents were tested to determine their shear bond strength to three different metals: amalgam, gold and base metal.

In this study, Prisma Universal Bond 3 presented lower bond strength to noble metal and base metal alloys than the bond strength claimed by the manufacturers. They claimed that the bond strength of sandblasted precious metal to Prisma was 13.45 MPa and non-precious metal was 17.28 MPa. This study did not support this claim. The investigators found that the mean bond strength of Prisma to gold was 7.57 MPa  $\pm 1.29$  and for base metal was 8.73  $\pm 1.86$ .

The bond strength of Prisma to amalgam (7.5 MPa  $\pm 1.41$ ) was similar to the manufacturers claim (8.32

Table I - Bond Strength

		N	Mean (MPa)	Std Dev
Prisma Universal Bond 3	Amalgam	10	7.51	+/-1.41
	Gold	10	7.57	+/-1.29
	Base Metal	10	8.73	+/-1.86
Scotchbond Multi-Purpose	Amalgam	20	10.51	+/-3.55
	Gold	10	10.12	+/-1.59
	Base Metal	10	13.37	+/-1.58
All-Bond 2	Amalgam	10	6.38	+/-1.59
	Gold	10	6.78	+/-1.44
	Base Metal	10	11.17	+/-1.79

Table II - Newman-Keuls Multiple Comparison

Bond Agents	N	Mean
Prisma Universal Bond 3	30	7.938
All-bond 2	30	8.1107
Scotchbond Multi-Purpose	40	11.127
<b>Metals</b>		
Amalgam	40	8.727
Gold	30	8.158
Base Metal	30	11.091

\*Means connected by vertical lines are not significantly difference  $p < 0.05$

MPa).

Shear bond strength of Prisma Universal Bond 3 was not significantly different ( $p>0.05$ ) among the metals tested.

The mean bond strength to base metal found for Scotchbond Multi-Purpose ( $13.37 \text{ MPa} \pm 1.58$ ) was higher than the bond strength found for All Bond ( $11.17 \pm 1.79$ ) and Prisma ( $8.73 \text{ MPa} \pm 1.86$ ). Scotchbond had also the highest bond strength to amalgam and gold.

All-Bond system presented higher bond strength to base metal than to gold and amalgam. Still the bond strength of All-Bond to base metal was much lower than the bond strength reported in a study by Kanca<sup>4</sup>. In this study, the author used 90 psi pressure to air abrade the metal and then stored the specimens in water for only 24 hours. The fact that this study used less pressure for air abrasion (50 psi for five seconds), stored the specimens for 14 days, and then subjected them to thermal cycling may have contributed to the lower bond strengths. Also, the bond strength of All-Bond to sandblasted noble metal alloy was approximately three times lower ( $6.78 \text{ MPa} \pm 1.44$ ) than the bond strength to sandblasted precious alloy reported by Suh<sup>9</sup> ( $27.79 - 31.41 \text{ MPa}$ ). However, in that study the author tested the specimens at two and 24 hours after bonding and did not thermocycle the specimens prior to testing.

In this study, Scotchbond Multi-Purpose exhibited the highest bond strength to the three different metals tested. However, the coefficient of variation presented for amalgam/Scotchbond groups (34%) was higher than for the other groups, but the variances within the amalgam/Scotchbond groups were homogeneous at 0.01 level of significance. Thus, both groups were combined and resulted in the only group of 20 specimens.

Bond strength for All-Bond/amalgam ranged from 4.7 to 9.0 MPa. Roeder et al.<sup>7</sup> reported that the highest bond strength ( $5.5 - 8.4 \text{ MPa}$ ) was achieved with All-Bond applied to sandblasted amalgam surface and that thermocycling did not affect the bond strength of repaired amalgam.

Some laboratory studies<sup>1,2,6</sup> reported the use of an ultrasonic cleaner to clean the metal substrate after air

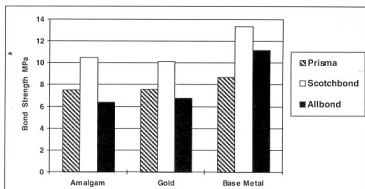


Figure 1 - Shear bond strength

abrasion. However, this study did not use ultrasonic cleaner because it is not achievable clinically since this study is using bonding agents that are recommended for repair of restoration that are fixed in the mouth. The clinical relevance of this study is that the bonding agents that are currently on the market may not be as effective as the manufacturer claim. It also may be possible that the different methodology may have influenced the bond strengths achieved in this study. If the noble metal had been tin plated, it may have had higher bond strength, but we have to consider that tin plating units are not frequently found in dental offices.

If it were necessary to select a bonding agent to repair a metal restoration, this author would choose SBMP to repair base metal restorations.

Considering the results of this study, it is indicated to use some type of mechanical retention whenever possible when bonding composite to metal restorations, and if air abrasive would be used, one might suggest increasing the pressure to 80 psi for 10 to 15 seconds since studies using this pressure achieved higher bond strengths.

This study did not investigate the influence of different surface treatments, longer storage time and higher abrasive pressure on the bond strength to metals, or whether if tin plating would improve the bond to precious alloy.

This study may be more valid than other studies using the same products because the specimens were thermocycled and stored for a longer period.

Future studies should evaluate different surface treatments as well as longer storage times.

## CONCLUSIONS

-Scotchbond exhibited the highest bond strengths to all metals. These strengths were significantly greater ( $p<0.05$ ) than those of All-Bond and Prisma Universal Bond 3. All-Bond and PUB 3 bond strengths were not significantly different ( $p=0.05$ ).

-All agents exhibited the highest bond strengths to base metal. These strengths were significantly different ( $p<0.05$ ) from bond strengths to amalgam and gold. Bond strengths to the latter metals were significantly different ( $p=0.05$ ).

-All the samples presented adhesive failure between metal/bonding agent interface.

## RESUMO

Este estudo avaliou a resistência ao cisalhamento de três sistemas adesivos universais: Prisma Universal Bond3, All-Bond2 e Scotchbond Multi Purpose com três ligas diferentes: Dispersalloy, Loadstar e Litecast B. A superfície metálica foi condicionada com uma lixa de Carbeto de Silício (600 grit), seguida de microjateamento (Óxido de alumínio 50 mm - 50 psi - 5 seg.). Todos os corpos de prova foram armazenados em água a 37°C por 48 horas, sendo a seguir termociclados entre temperaturas de 8°C e 48°C (2.500 ciclos). Na sequência à termociclagem, estes corpos de prova foram armazenados em água por 14 dias, sendo então submetidos aos testes de cisalhamento (Instron - velocidade de afastamento de 1 mm/min.). A carga onde ocorreu a ruptura foi registrada. As interfaces de ruptura foram examinadas com o auxílio de um microscópio (40X), visando determinar e avaliar os sítios onde ocorreram as falhas do processo adesivo.

Scotchbond Multi Purpose apresentou os mais altos resultados para resistência de união a todas as ligas. Esta resistência foi significativamente maior ( $p<0.05$ ) do que as obtidas com All-Bond2 e Prisma Universal Bond3, que foram semelhantes entre si ( $p=0.05$ ). Todos os agentes exibiram maior resistência de união com as ligas de metais básicos. Esses valores foram significativamente diferentes ( $p<0.05$ ) dos valores obtidos com amálgama e ouro. A resistência de união frente aos dois últimos foi semelhante ( $p=0.05$ ). Todos os corpos de prova apresentaram falhas adesivas na interface metal/agente de união.

**UNITERMOS:** Resistência de união; Adesivos; Metais.

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