COMPOSITE-TO-METAL BOND; THE EFFECT OF THE OPAQUE LAYER

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ABSTRACT

The purpose of this study was to evaluate, if the presence of the opaque resin as an intermediary layer between the veneering composite and the metal alloy has a weakening effect on the overall composite-to-metal bond.

In order to evaluate the effect of the opaque layer 15, pairs of gold rods were grit blasted, cleaned and primed with the mixed silane primer (MPTS / MPMS). A thin layer of 70-µm thickness of the opaque resins or the composite resins of Sinfony, Solidex, Pertac hybrid and Esthet.X was applied on the adhering surfaces of the rods before they were joined using Variolink II luting resin. All the bonded rods were stored dry for 24 hours. A Lloyd Universal Testing Machine was used to test the tensile bond strength at a crosshead speed of 1 mm / min. The debonded surfaces were observed under an Olympus Optical Microscope and the mode of failure was recorded. ANOVA and Tukey's test were used to determine significant differences between the groups.

There was no significant difference in the TBS of specimens prepared with the opaque resins when compared with the TBS of the control group or TBS of their relative composites. Most of the specimens showed a mixed mode of failure with the exception of Sinfony opaque, which showed cohesive failure. The opaque resin layer has no weakening effect on the overall composite-to-metal bond.

Key words: *Composite Resin, Opaque Resin, Mixed Silane Primer*

INTRODUCTION

The introduction of resin-based composite restorative materials had a major impact on the practice of restorative dentistry.[1]

Resins have been used as veneers for crown and bridge frameworks since 1940. Although the fabrication of resin veneered restorations is simple, fast and cheap [2,3,4] clinicians encountered many problems with the early generations such as discoloration, poor wear resistance and fluid leakage at the resin metal interface.[3,5,6]

The weak bond between composite resin and the metal alloys required the use of number of techniques to enhance the bond strength.[7,8,9,10] Most of these methods were time consuming, technique sensitive and restricted the choice of the alloys to certain non-noble metals. Furthermore, they still relied on micro-mechanical retention and microleakage remained a problem.[10]

During the last decade a major breakthrough was made in the development of new metal-resin bonding techniques that resulted in a chemical bonding between resin and metal.[11,12,13] Bonding with base metal alloys is no longer a problem with the use of 4-META or MDP monomer based resins.[14,15] Recently new types of thiol-containing functional monomers have been used successfully in improving the adhesion of composite resins to noble and precious metals.[16,17,18] They offer a simple and effective method of bonding with the possibility of their use in intraoral repairs. Mixed silane primers (MPTS/MPMS) are composed from methacryloxy propyl trimethoxy silane (δ-MPTS) and 3- mercapto propyl trimethoxy silane (3-MPMS). The MPMS provides the –SH or mercapto group that facilitates the binding with noble and precious alloys while

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the MPTS provides the methacrylate group for copolymerisation with composite resins.[19]

Opaque resins are usually applied as a thin layer underneath the veneering composite on the metal surface to mask the colour of the underlying metal.[20] The presence of titanium dioxide, zirconium dioxide and aluminum trioxide within the composition of the opaque resins render them effective in hiding the colour of the underlying metal.[21]

However, the addition of large amount of these metal oxides to the resin may adversely affect its handling and physical properties.

Little has been written in the literature about the opaque resins. Most of what has been written was about the effect of filler and monomer composition of prepared opaque resins on the physical and mechanical properties of the opaque layer itself and the influence of this on the bond strength with the metal alloy.[22-30] But none of the previous works discussed whether the presence of the opaque resin as an intermediary layer between the veneering composite and the metal could be a weak link in the composite-to-metal bond.

This study tests the possible effect of the opaque resin layer on the bond between the veneering composite and a precious metal alloy.

MATERIALS AND METHODS

Gold alloy rods of 15 mm in length and 4 mm in diameter were used. The adhering surfaces of the rods were ground with 600- grit silicon carbide papers to produce a relatively flat surface. The adhering surfaces were then grit blasted with 50 µm aluminium oxide for 10 seconds at a pressure of 4 bar with the nozzle positioned 5 mm away from the alloy surface. The rods were finally cleaned with acetone in an ultrasonic bath for 10 minutes and left to dry. 14 pairs of rods were used for Exp 1-10

The mixed silane primer (MPTS/MPMS) was prepared by mixing the following constituents by volume MPTS(1.25%), MPMS(1.25%), Ethanol(95%) and Acetic acid (2.5%). After mixing the different components, the solution was stored in the fridge and used within one month of preparation. The following procedure was used for the application of the mixed silane primer as previously described in other studies[31,32]

Application of a thin layer by brush, air drying for 60 seconds, hot drying for 15 seconds, rinsing with hot water (70°C) for 15 seconds, and hot drying for 15 seconds.

After the application of the mixed silane primer a thin layer of the opaque resin was applied carefully on the adhering surface of the primed rods using a plastic instrument. The alignment jig (figure 1) was used to ensure an approximate thickness of 70 µm of the opaque layer on the surface of the prepared and primed rods. The opaque layer was cured using an Espe Elipar® Trilight curing unit for a minimum of 40 seconds. The two rods were then bonded with the Variolink II luting cement using a precalibrated alignment apparatus with a total of 210 µm gap width. The Variolink II was cured from 4 points for 30 seconds each using Espe Elipar® Trilight and 3M Curing light XL 3000 curing units. Table 1 shows summary of the used materials. Figure 2 shows a diagrammatic representation of experiments 3-6. For experiments 7-10 the same procedure was applied as before with the exception that the corresponding composite resin was applied instead of the opaque resin for each tested material as shown in figure 3. Table 2 shows detailed summary of experiments 1-10. A Lloyd Universal Testing Machine (Lloyd Instruments Ltd. Farham. U.K) was used to test the tensile bond strength of the bonded specimens for experiments 1-10.

The bonded rods were held vertically with a specially designed gripping apparatus within the testing machine as shown in figure 4.

The bonded rods were subjected to a tensile force at a crosshead speed of 1 mm/min and the tensile bond strength values were recorded in MPa.

The debonded surfaces were observed by Olympus optical microscope (Olympus TL, Olympus optical Co., Japan) to record the mode of failure. Failure was recorded as adhesive if it occurred at the resin metal interface, or cohesive if it occurred through the resin (luting resin, composite resin, opaque resin), or mixed if it was a combination of both adhesive and cohesive.

Statistical analysis

Individual tensile bond strengths (MPa) and the mode of failure (A, C, M) readings were calculated and tabulated. The mean and the standard deviation were

TABLE 1: SUMMARY OF THE USED MATERIALS

Material	Identification	Manufacturers
Alloy	Golden multibond(Au 86.8, Pt 11.8 PL)	Metalor Co., NeuchatelSwitzerland
Laboratory Composites	• Sinfony – Composite (A4) – Opaque (A4O) • Solidex· – Composite (A4) – Opaque (A4O)	ESPE. Dental-medizin GmbH & Co., KG. Seefeld. Germany Shofu Inc., Kyoto, Japan
Clinical Composites	• Pertac Hybrid – Composite (DB=A4) – Opaque (DBO=A4O) • Esthet.X – Composite (A4) – Opaque (A4O)	ESPE. Dental-medizin GmbH & Co., KG. Seefeld. Germany Dentsply Caulk, Milford, USA
Metal Primer	• Mixed Silane Primer – Bifunctional monomer (MPTS/MPMS) – Solvent (Ethanol) – Catalyst (Acetic Acid)	Sigma chemical Co., St. Louis. USA Gental Medical, Tockwith, York. UK UKBDH Laboratory Supplies, Poole, UK
Composite luting cement	Variolink II cement (high viscosity	Vivadent. Schaan, Liechtenstein

MPTS/MPMS= Methacryloxy propyl trimethoxy silane/Mercapto propyl trimethoxy silane

TABLE 2: DETAILS OF EXP. 1-10

Part II	Gap width 210 um	Gold alloy	No primer	MPTS/ MPMS/ primer	Sinfony Co Op	Solidex Co Op	Pertac H Co Op	Esthet.X Co Op	Variolink
Exp. 1	√	√	√						√
Exp. 2	√	√		√					√
Exp. 3	√	√		√	√				√
Exp. 4	√	√		√		√			√
Exp. 5	√	√		√			√		√
Exp. 6	√	√		√				√	√
Exp. 7	√	√		√	√				√
Exp. 8	√	√		√		√			√
Exp. 9	√	√		√			√		√
Exp. 10	√	√		√				√	√

Co = Composite **Op =** Opaque **n** =15

recorded. Statistical analysis was carried out using Micro soft Excel computer software. Data were compared by one way analysis of variance (ANOVA) with the tensile bond strength as the dependent variable. When ANOVA showed significance, the Tukey's test from Minitab with $P < 0.05$ was employed to identify differences between the groups.

RESULTS

The means of the TBS and the standard deviation as a function of the resin are summarised in Table 3 and presented graphically in Figure 5.

One way analysis of variance revealed that the tensile bond strength was significantly affected by the

TABLE 3: SUMMARY OF THE RESULTS OF EXP. 1-10

Part II	n	Gap width (μm)	Mean TBS(MPa)	SD (+/-MPa)	Tukey's group*	Mode of failure A	C	M
Exp. 1Au-no primer	15	210	3.78	0.93	a	Adhesive failure with signs of porosity		
Exp. 2 Au- MPTS/MPMS	15	210	24.02	8.44	b c d	–	1	14
Exp. 3 Au-Sinfony (O)	15	210	25.37	4.51	b c d	–	15	–
Exp. 4 Au-Solidex (O)	15	210	25.07	5.97	b c d	1	2	12
Exp. 5 Au-Pertac (O)	15	210	22.33	8.14	b c d	–	–	15
Exp. 6 Au-Esthet.X (O)	15	210	25.60	11.67	b c d	–	1	14
Exp. 7 Au- Sinfony	15	210	29.39	7.59	d	–	10	5
Exp. 8 Au-Solidex	15	210	16.67	4.56	b	–	13	2
Exp. 9 Au- Pertac	15	210	18.72	7.61	b c	1	–	14
Exp. 10 Au- Esthet.X	15	210	26.93	9.80	c d	–	–	15

TBS= Tensile bond strength **SD=** Standard deviation
* Different letters indicate that the values are statistically different $P < 0.05$
A = Adhesive failure **C =** Cohesive failure **M =** Mixed failure

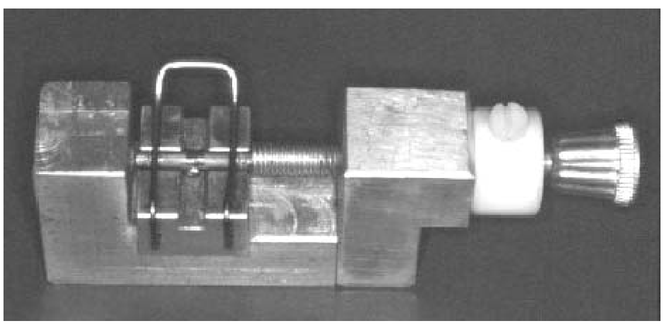

Fig 1: Alignment apparatus with the bonded gold rods

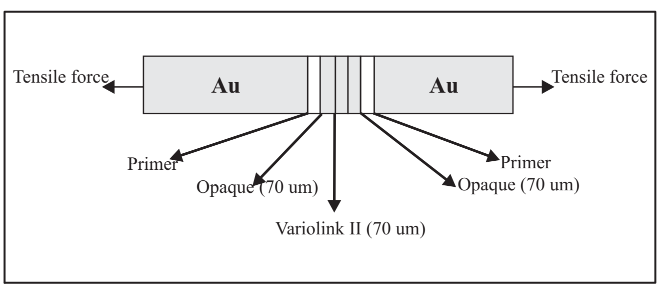

Fig 2: Diagrammatic representation of Exp. 3-6

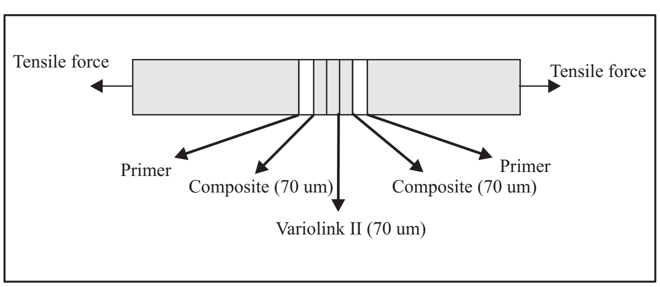

Fig 3: Diagrammatic presentation of Exp. 7-10

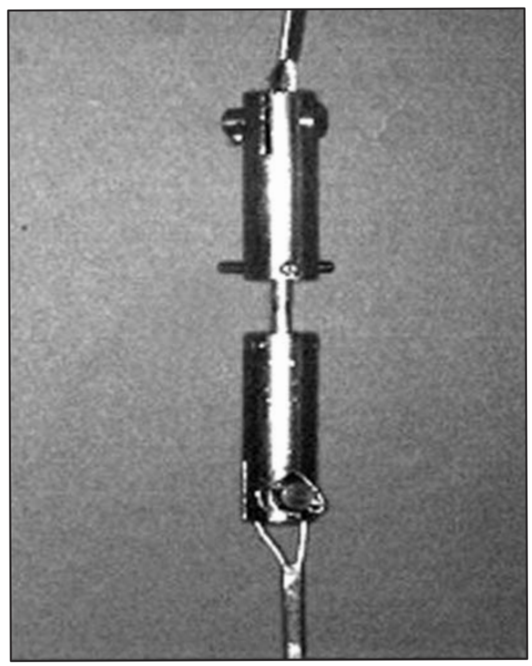

Fig 4: Specimens held vertically in the testing machine

use of the metal primer $P < 0.05$. The lowest tensile bond strength was recorded in Exp.1 (3.78 MPa) when no primer was used to enhance the bond between the gold alloy and the resin. The mode of failure was adhesive between the resin and the alloy. The low value of the tensile bond strength with the adhesive mode of failure indicates the weakness of the bond between the resin and gold alloy. A TBS value of 24.02 MPa was recorded when the MPTS/MPMS primer was

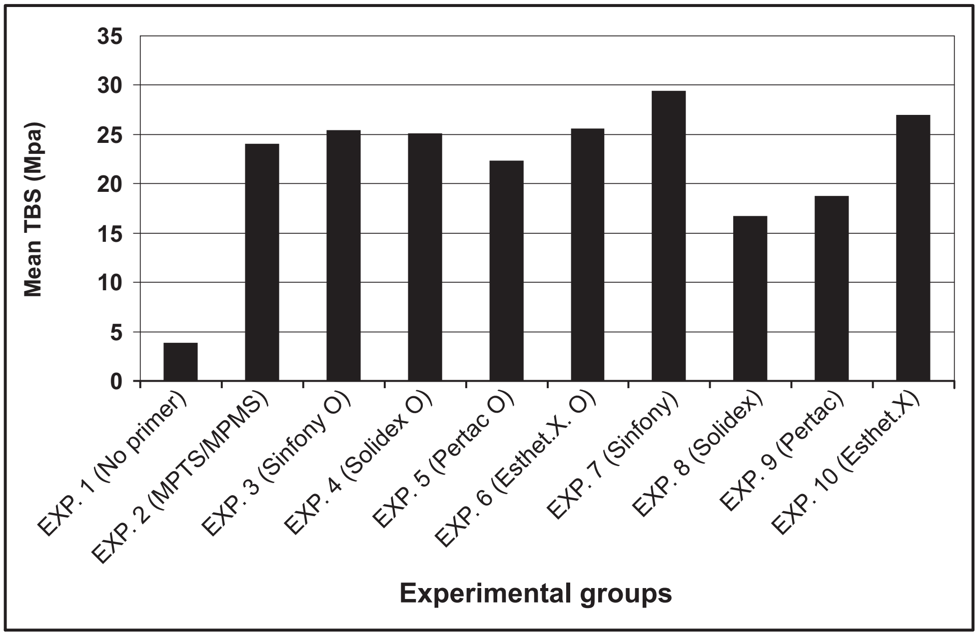

Fig 5: Mean tensile bond strength of the tested opaque and composite resins

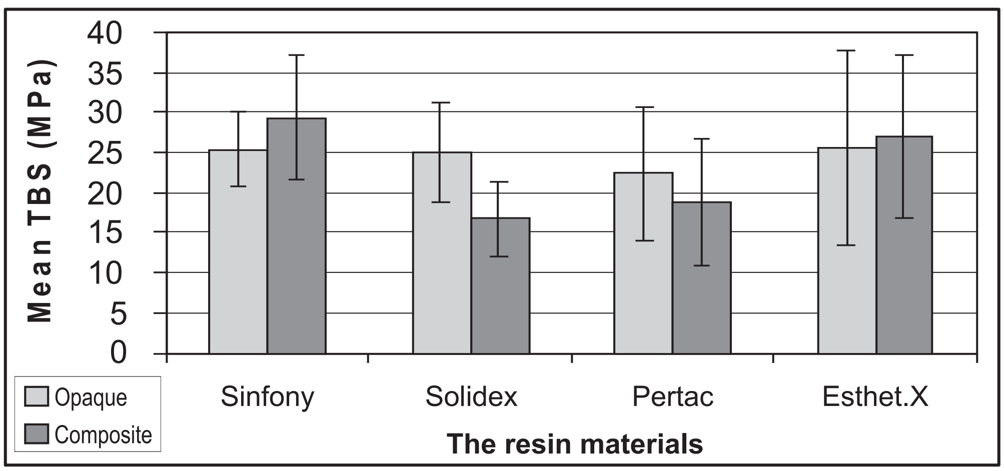

Fig 6: Comparison of the mean TBS values of the opaque and composite resins

used (Exp. 2). The use of this primer has shifted the mode of failure from purely adhesive in Exp. 1 to a more favourable cohesive or mixed failure.

The primed gold alloys in Exp. 2, which were joined with the Variolink II luting cement without any opaque or composite served as a control.

The tensile bond strength values of Exp. 3-6, which were prepared using the opaque resins, ranged between 22.33-25.60 MPa. The results of one way analysis of variance and Tukey's test showed no significant difference between these values and the TBS of the control (24.02 MPa). Regarding the mode of failure, most of the debonded specimens showed a predominantly mixed mode of failure, which was consistent with the mode of failure in the control group with the exception of Sinfony, which showed cohesive failure in all the specimens.

When the composite resin was used in the preparation of the rods for Exp. 7-10, the tensile bond strength values ranged from 16.67- 29.39 MPa. The highest value was recorded with Sinfony and the lowest value was recorded with Solidex.

The tensile bond strength values of the opaque resins were not significantly different from the tensile bond strength values of their composite resins as shown in Figure 6.

Regarding the mode of failure of Exp. 7-10, the specimens showed a mixed mode of failure similar to their opaque resins with the exception of Solidex in which the mode of failure was predominantly cohesive.

DISCUSSION

Noble metal alloys are usually preferred in the fabrication of casted restorations, but adhesion of resins to noble and precious alloys has been known to be a problem. The development of varying adhesive systems for noble alloys has increased the use of these alloys in the fabrication of casted restorations substantially. [18] A simple and effective method that has been proved to promote the chemical adhesion of resin to metal alloys is the use of metal primers. [33,34]The use of theses primers as adhesion promoters is promising; the procedure offers the advantages of being simple and can be used intraorally.

In this study the mixed silane primer (MPTS/MPMS) was used to enhance the bond between the resin and the gold rod. Anagnostopoulos, Eliades and Palaghias [35] and Matinlinna, Lassila and Vallittu[36,37] reported the effectiveness of silane primers containing ä-methacryloxy propyl trimethoxy silane (δ-MPTS) as adhesion promoters between composite resins and the metal alloys. 3- mercapto propyl trimethoxy silane (3-MPMS) which has a mercapto group as an organo-functional group will probably bond to the metal surface.[19]

Umemoto and Kurata[19] Sudsangium[31] and Althwaini[32] have reported the successful use of the mixed silane primer (MPTS/MPMS) in enhancing the bond strength of composite resin to different precious and non-precious alloys. The MPMS provides the –SH group that facilitates the binding with noble and precious alloys, while the MPTS provides the methacrylate group for copolymerisation with composite resins.

The procedure that has been followed for the application of the MPTS/MPMS (brush application, heat treatment and rinsing with hot water) has been reported to improve the tensile bond strength values of specimens treated with the silane primer.[38]

Tensile bond strength testing was used in this study depending on the results of previous work done by van Noort *et al* [39] and Della Bona and van Noort [40] who found that the calculated shear bond strength is not a true reflection of the stresses at the adhesive interface due to the non-uniformity of the stress distribution. They reported that the tensile bond testing might provide a more representative measurement of the adhesive bond. The MPTS/MPMS primer was used within 1 month of its preparation since it was found that it would still be effective as an adhesion promoter within this period. [31]

The TBS values of the four opaque resins revealed that there was no significant difference between the different opaque resins (Exp. 3-6) and the control (Exp. 2).

Regarding the mode of failure, all the opaque resins showed a mixed mode of failure with the exception of the Sinfony, which showed consistent cohesive failure through the opaque layer. This may suggest a weakness in the material; nevertheless, the TBS values of the Sinfony opaque were high and not significantly different from the other opaque resins.

The problem with the mixed failure is the difficulty in determining the weakest point because the failure could start at the metal resin interface and continue through the material or it could start cohesively through the resin and end as an adhesive failure. SEM or even more sophisticated methods such a SIMS can be helpful in revealing the weakest point that started the failure.

In comparing the TBS of each opaque with its composite there was no significant difference. Both Solidex and Pertac hybrid composite showed unexpected lower TBS values than their relative opaque resins. For the Solidex there was also a change in the mode of failure from a mixed failure to cohesive failure which may indicate a weakness in the material. Touati and Aidan [41] mentioned in their study that Solidex has a low flexural strength (75 MPa) compared with the flexural strength of other current laboratory composites (120-160 MPa) which to some extent can explain these findings. However, it does not explain the higher value of the Solidex opaque resin unless there is a distinct difference in the chemical composition of both which was not obvious in the manufacturer's literature. The Pertac hybrid specimens were prepared nearly at the end of one month of the primer preparation, when it is possible that the primer may have started to lose its efficacy, which could explain these low results. Further investigations of the flexural strength and the fracture toughness of these materials may explain these results, but surprisingly this information is not usually provided by the manufacturers and not easily found in the literature.

It was found from a simple subjective assessment of the data that there was no correlation between the mode of failure and the TBS values, as mentioned previously by Petridis *et al.* [42]

Although it was found from the previous results that the opaque layer has no obvious weakening effect on the overall composite-to-metal bond, this study depended on one test which is the tensile bond test and the whole system may behave differently in the mouth with the presence of other factors such as saliva, thermal changes and cyclic loading.

CONCLUSION

The opaque resin layer has no weakening effect on the overall composite-to-metal bond.

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