

# AN EXPERIMENTAL STUDY ON THE BOND STRENGTH OF THE LIGHT ACTIVATED COMPOSITE RESIN BONDED TO THE ETCHED PORCELAIN

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## I. INTRODUCTION

The porcelain — fused — to metal (PFM) system has been used by the profession for close to few decades. During this time improvements have occurred in the physical properties and composition of alloys, porcelain, and investments, in the ways of handling of the materials, and in design. Despite of these improvements porcelain fractures, cracking and flaking off the coping are still common occurrences.<sup>2)</sup> Fractures of PFM restorations may be attributed to single or multiple deficiencies. Potential causes of failure include lack of ceramo — metal adherence, thermal contraction incompatibility, design and procedure errors, malocclusion, poor handling, or faulty design. The ideal way of correcting these problems would be to replace the restorations<sup>1)</sup> However, this may not be the practical approach, specially when the failure is part of a long span restorations. Compensating for above disadvantage, there are two methods which were clinical — laboratory procedures and clinical procedure. Gelibman, Biber and Dent<sup>9,14)</sup> reported the salvaging the broken porcelain fused to metal partial prosthesis in clinical — laboratory procedures such as PFM overcasting and PFM pin retained casting. One clinical

procedure was the composite resin bonded to porcelain as a intraoral repair method. Previous many studies manifested that the use of silane increased the bond strength of composite resin to etched porcelain and decreased the marginal leakage.

In 1960, silane coupling agents were introduced commercially in glass — reinforced plastics. Bowen (1962) used these materials in the development of composite resins that were reported to the dental profession in 1963.<sup>38,40)</sup> Recently, silane coupling agents are used for bonded porcelain teeth to acrylic resin denture resin denture,<sup>29,30,35,36,42)</sup> direct bonding of orthodontic brackets<sup>39)</sup>; restoration of cavity<sup>5)</sup>; and porcelain laminate veneer<sup>3,6,8,20,21,23,24,28,31)</sup> technique. Especially, good results were achieved by using a silane coupling agent in the repair of fractured porcelain. Newberg and Pameijer<sup>38)</sup> reported that a system of bonding composite resins to dental porcelain with a silane solution produced a reliable bond. This technique might be a clinical solution to intraoral repair of fractured or chipped porcelain restoration. Highton, Caputo and Matyas<sup>17)</sup> compared the effectiveness of two new porcelain repair systems which used coupling agents. Rehany, Zalkind and Revah<sup>47)</sup> reported the repair of fractured porcelain jacket crown with a composite resin using a

silane coupling agent.

Previous studies had shown that etched porcelain with a silane coupling agent enhanced bonding strength of porcelain – composite resin. Recently, this method is widely used in porcelain laminate veneer technique which were physical and chemical bonding mechanism. Porcelain laminate veneer technique is indicated for repairing ceramo – metal fixed bridge, masking discolorations and labial irregularities, closing diastemas, custom – shaped porcelain orthodontic brackets, and for porcelain inlay. Calamia et al.<sup>7)</sup> showed that each porcelain had a specific concentrations and time of hydrofluoric acid etching for maximum bond. Calamia and Simonson<sup>6,53)</sup> reported the effect of coupling agents on bond strength of etched porcelain using a etching solution containing hydrofluoric and sulfuric acid and tensile bond strength of composite resin to porcelain were significantly increased by etched porcelain using a solution containing 7.5% hydrochloric acid. Hsu, Jones and Horn<sup>23,25,31)</sup> reported that porcelain samples were etched with a hydrofluoric acid substitute, Stripit. Calamia, Vaioyanathan and Hamburg<sup>8)</sup> studies showed that etching porcelain with a commercial hydrofluoric acid substitute enhanced tensile and shear strength of porcelain com-

posite resin bond with a Dicor<sup>TM</sup> castable ceramic. Hobo and Iwata<sup>21)</sup> studied a new laminate veneer technique using a castable apatite ceramic material, CeraPear1 and 2N hydrochloric acid was an effective etching solution for the apatite during 15 minutes.

There were many contributing factors for bonding strength of porcelain – composite resin: etching time and kinds of porcelain etching solution, kinds of composite resin and whether or not a silane coupling agent was used. When there was a part of porcelain fracture without metal exposure in PFM crown, An aesthetic repair using a composite resin could be done as an intraoral method. The purpose of this study was to show improved clinical repair method in PFM crown fracture by comparing the tensile strength in the various bonding treatment of porcelain – composite resin.

## II. METHODS AND MATERIALS

In this study, materials were employed as shown in Table 1.

i) preparation of substrate and surface treatment

The lingual surfaces of 135 upper central

Table 1. Materials

A. Bond strength measurement	B. Porcelain etching solution	C. Porcelain sample
1. Silane coupling agent (Scotch-prime)*	1. 5% HF solution****	Vacuum fired Porcelain
2. Bonding agent (Scotchbond)**	2. Porcelain etching	denture teeth*****
3. Composite resin (Heliosit)***	gel (Excelco)*****	

\* Scotch-Prime, 3M Company Dental Products (U.S.A.)

\*\* Scotchbond, 3M Company Dental Products (U.S.A.)

\*\*\* Heliosit, Vivadent (LIECHENSTEIN)

\*\*\*\* Hydrofluoric acid. Shinyo Pure Chemicals Co. (JAPAN)

\*\*\*\*\* Porcelain etching gel, Excelco International Inc. (U.S.A.)

\*\*\*\*\* Vacuum fired porcelain denture teeth, G.C.'s Dental Industrial Corp. (U.S.A.)

incisor porcelain denture teeth were embedded in a block of cold curing acrylic resin. The labial surfaces were ground flat to provide surface greater than 3mm diameter; then they were smoothed on a 400Cw (grit) silicone carbide paper on running water after the surfaces were roughened with a high speed round diamond point to remove any penetrating scoring that might provide mechanical retention. And all porcelain samples were cleaned with ultrasonic cleaner for 10 minutes.

ii) Bond strength measurement

A total of 135 specimens were prepared and assigned to the following groups (Table 2): The specimen in group A (60 teeth) was etched with 5% HF etching solution. In A group, subgroup a<sub>1</sub> (20 teeth) was treated with silane coupling agent and composite resin, subgroup a<sub>2</sub> (20 teeth) was treated with bonding agent and composite resin and subgroup a<sub>3</sub> (20 teeth) was treated with silane coupling agent, bonding agent and composite resin at different etching time (2.5, 5, 10, 20 min). The specimen in group B was prepared like those in group A, except group B with porcelain etching gel (Excelco) and control

group with no etching.

The bonding area was defined by covering approximately the center of each porcelain sample with PVC (poly vinyl chloride) adhesive tape which a hole had been punched. Bonding agent (scotch bond) and/or silane coupling agent (scotch – prime) was polymerized on the porcelain surface. A button of composite resin with 3mm diameter was then fabricated.

Tensile test method (pressure test):

The threaded Remanium orthodontic wire (Dentaram) 30 mm long and 1.00 mm diameter was placed perpendicularly to the center of the sample. And then, according to the manufacture's instructions, bonding agent and/or silane coupling agent was mixed and placed on the porcelain surface. The visible light composite resin was added and built up around the Remanium orthodontic wire in the shape of a cone 6 mm high, in three horizontal layers with visible light composite resin over a 3 mm diameter hole in the PVC tape. Each layer of the composite resin was cured for 40 seconds by directing high intensity fiberoptic light (Estilux) from mesial, distal, anterior and posterior directions for

Table 2. Number of specimen of each group

Group	Etching	Treatment	Specimen No.				
			Etching time (min.)	C (Control)	2.5	5	10
A	5% HF solution	a <sub>1</sub> (S+V)	5 (C <sub>1</sub> )	5	5	5	5
		a <sub>2</sub> (B+V)	5 (C <sub>2</sub> )	5	5	5	5
		a <sub>3</sub> (S+B+V)	5 (C <sub>3</sub> )	5	5	5	5
B	Porcelain etching gel (Excelco)	b <sub>1</sub> (S+V)		5	5	5	5
		b <sub>2</sub> (B+V)		5	5	5	5
		b <sub>3</sub> (S+B+V)		5	5	5	5

S: Silane coupling agent (Scotch-prime)

B: Bonding agent (Scotch bond)

V: Visible light composite resin (Heliosit)

10 seconds.

After polymerization, the tape was removed, leaving only the resin in contact with the porcelain. Bond strength were determined after storage in 37°C water for 24 hours using a Shimadzu universal mechanical testing machine at crosshead speed of 2 mm/min. The bonded specimens were clamped to the crosshead of the testing machine, one end of Remanium orthodontic wire attached to the load cell could be pulled off in tension. (Fig. 1). The specimen were tested by the application of a point force load on the composite resin button at its junction with the porcelain. The bond strength calculated as a fracture load divided by bonded surface area on 0.3 mm diameter was evaluated in tension.

### iii) Scanning electron microscope (SEM) analysis

To investigate the etching pattern according to porcelain etching solution and etching time,

unetched porcelain sample (1 sample) and 8 porcelain samples which were etched by 5% HF solution (4 samples) & Excelco (4 samples) at different etching time (2.5, 5, 10, 20 min.) were examined by SEM. Selected porcelain samples were mounted on aluminum stubs. Nine porcelain sample (0.5x 0.2x 0.2 cm) were made conductively by coating with gold paladium in a sputter coater (Go Ko, IB - 3 Ion Coater, Eiko Engineering Co., Ltd. Japan).

Sputtering conditions were as follows:

Vacuum (0.1 Torr)

Sputtering time fixed at a layer thickness of 100Å°

Sputtering voltage of 30 kv

Current of 6 mA

All porcelain samples were viewed in a SEM (super - IIIA, ISI International Scientific Instruments, Japan) at magnification up to 500 X and were taken with polaroid type 667 film.

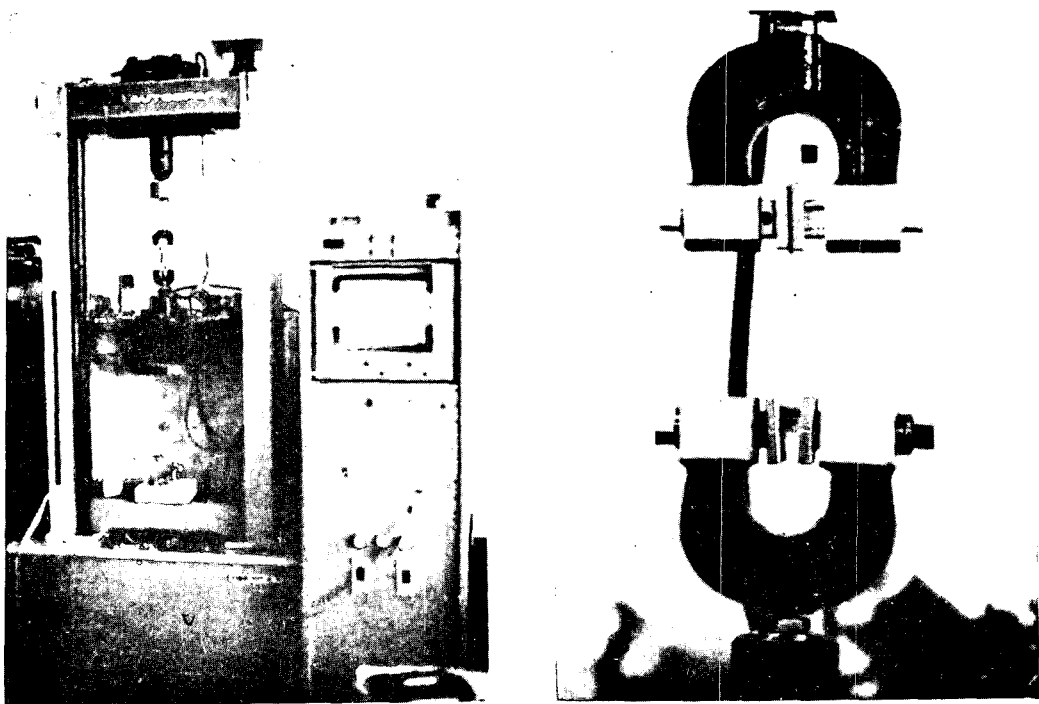


Fig. 1. Specimens clamped into testing machine to measure bond strength in tension

### III. RESULTS

For the tensile bond testing, the mean values and standard deviations for all groups were reported in Table 3.

Statistical analysis between experimental group (etched porcelain) and control group was highly significant. (Table 4.)

Etched porcelain samples were higher than control group in bond strength.

Two way analysis of variance results revealed statistical significance among porcelain treatment and among etching time in the group etched with 5% HF solution and in the group etched with Excelco. (Table 5).

Both in the group etched with 5% HF solution and in the group etched with Excelco, the bond

Table 3. Bond strength between porcelain and composite resin

Group	Etching	Etching time (min) Treatment	Bond Strength mean (kg/cm <sup>2</sup> )							
			C (control)	2.5	5	10	20	Total mean	S.D.	
A	5% HF	a <sub>1</sub> (S+V)	53.3 ± 3.80 (C <sub>1</sub> )	133.3 ± 1.90	135.5 ± 2.55	140.0 ± 3.26	143.3 ± 3.04	138.0	4.8	F: 21.12 P < 0.01
		a <sub>2</sub> (B+V)	40.4 ± 1.56 (C <sub>2</sub> )	115.5 ± 3.71	122.2 ± 2.69	125.5 ± 3.06	134.4 ± 4.36	124.4	7.86	
		a <sub>3</sub> (S+B+V)	86.6 ± 1.66 (C <sub>3</sub> )	175.5 ± 5.40	185.5 ± 3.50	187.7 ± 4.38	191.1 ± 3.03	185.0	6.71	
F: 235.21 P < 0.001										
B	Porcelain etching gel (Excelco)	b <sub>1</sub> (S+V)	53.3 ± 3.80 (C <sub>1</sub> )	127.7 ± 4.63	140.0 ± 5.10	157.7 ± 2.86	156.6 ± 4.27	145.5	14.37	F: 32.05 P < 0.001
		b <sub>2</sub> (B+V)	40.4 ± 1.56 (C <sub>2</sub> )	106.6 ± 3.39	124.4 ± 2.98	138.8 ± 3.62	137.7 ± 2.33	126.9	15.02	
		b <sub>3</sub> (S+B+V)	86.6 ± 1.66 (C <sub>3</sub> )	183.3 ± 1.92	192.2 ± 2.08	227.7 ± 5.17	222.2 ± 3.76	206.4	21.90	
F: 258.90 P < 0.001										

S: Silane coupling agent (Scotch-prime)

B: Bonding agent (Scotch bond)

V: Visible light composite resin (Heliosit)

Table 4. T-test between total experimental group (etched porcelain) and control group

Group	T value	P value
a <sub>1</sub> - c <sub>1</sub>	37.76	P < 0.001
b <sub>1</sub> - c <sub>1</sub>	12.85	P < 0.05
a <sub>2</sub> - c <sub>2</sub>	21.48	P < 0.001
b <sub>2</sub> - c <sub>2</sub>	11.57	P < 0.05
a <sub>3</sub> - c <sub>3</sub>	29.32	P < 0.001
b <sub>3</sub> - c <sub>3</sub>	10.93	P < 0.05

Table 5. Two way analysis of variance results in the group etched with 5% HF solution and in the group etched with Excelco

Scoure	DF		F ratio		P value	
	A	B	A	B	A	B
Treatment	2	2	235.21	258.90	P < 0.001	P < 0.001
Time	3	3	21.12	32.05	P < 0.01	P < 0.001

strength in relation to the lapse of etching time increased 5 minutes, but had no great increase after 5 minutes. (Fig. 2).

Bond strength of etched porcelain treated with silane coupling agent and bonding agent was highest and then, the group of treatment with silane coupling agent and the group of treatment with bonding agent in order. (Fig. 3).

In difference of bond strength between the group etched with Excelco and the group etched with 5% HF solution. The group treated with bonding agent did not show statistical significance ( $p > 0.5$ ). But the group treated with silane coupling agent and the group treated with silane coupling agent and bonding agent showed statistical significance ( $p < 0.05$ ,  $p < 0.001$ ).

SEM examination of etched porcelain group revealed that surface configuration varied with etching time and porcelain etching solution. The porcelain etched with Excelco demonstrated a relatively more porous surface texture compared



Fig. 2. Bond strength related to etching time in the group etched with 5% HF solution and the group etched with Excelco.

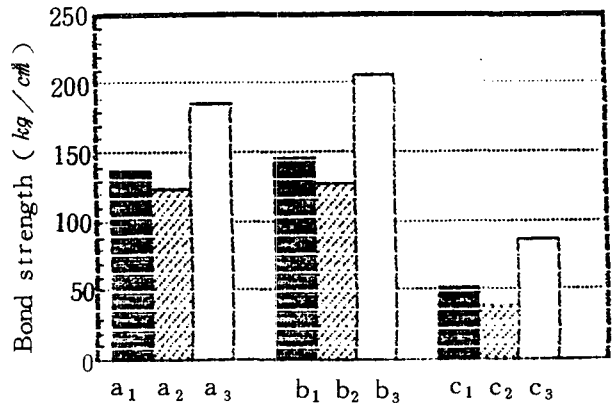


Fig. 3. Comparison of mean tensile strength according to the difference of treatment method.

Table 6. Statistical multiple comparisons test between the group etched with 5% HF solution and the group etched with Excelco

	a <sub>1</sub>	a <sub>2</sub>	a <sub>3</sub>	b <sub>1</sub>	b <sub>2</sub>	b <sub>3</sub>
a <sub>1</sub>	—					
a <sub>2</sub>	S*	—				
a <sub>3</sub>	S*	S*	—			
b <sub>1</sub>	S*	S*	S*	—		
b <sub>2</sub>	S	NS	S*	S*	—	
b <sub>3</sub>	S*	S*	S*	S*	S*	—

S: significant ( $P < 0.05$ )

S\*: very significant ( $P < 0.001$ )

NS: not significant ( $P > 0.5$ )

with the porcelain etched with 5% HF solution. An amorphous microstructure containing numerous large porosities was noted in Excelco. (Fig. 5). The surface was characterized by small porous and intact islands on relatively flat surface, suggesting less surface degradation in 5% HF solution than in Excelco. (Fig. 6).

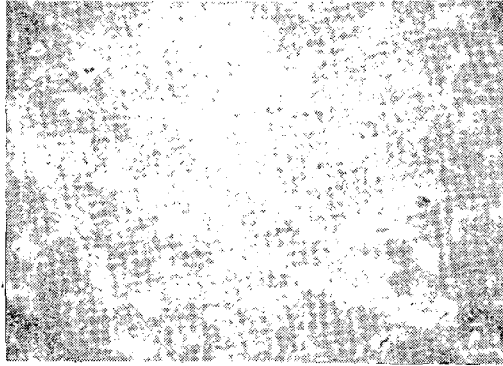
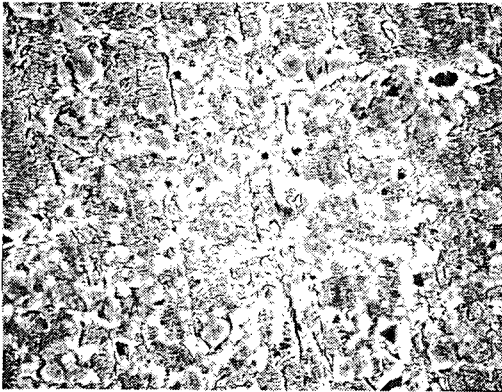
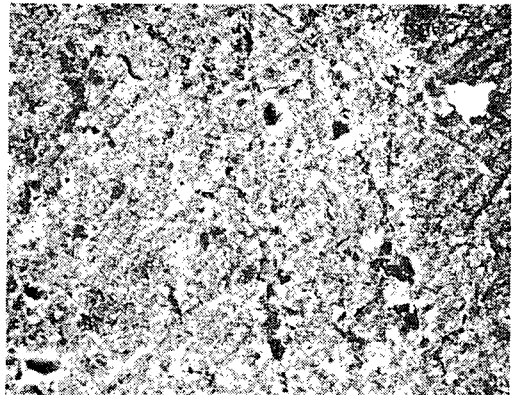


Fig. 4. SEM of porcelain sample at 500 magnification on the control group.



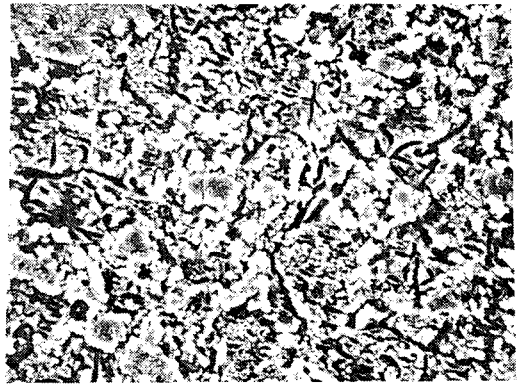
2.5 min. etch



5 min. etch

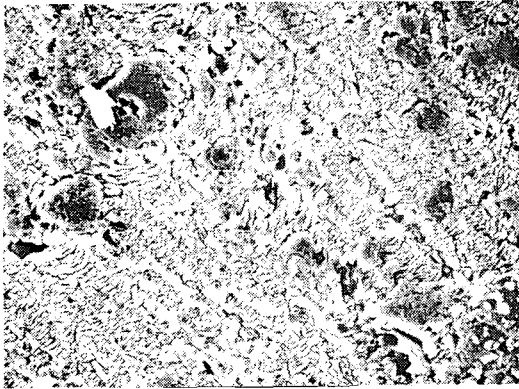


10 min. etch

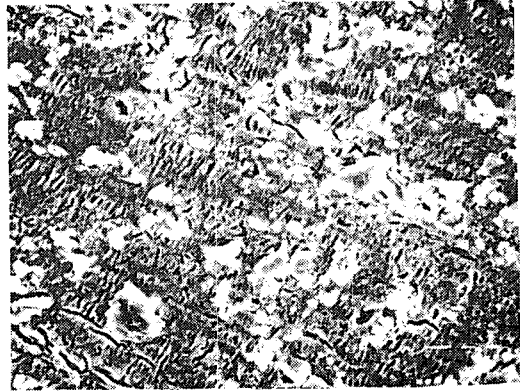


20 min. etch

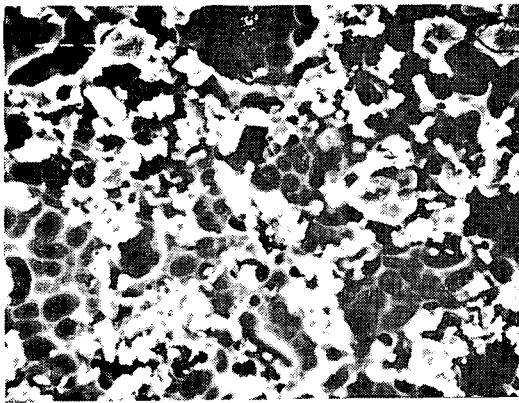
Fig. 5. SEM of porcelain sample at 500 magnification etched with a 5% HF solution.



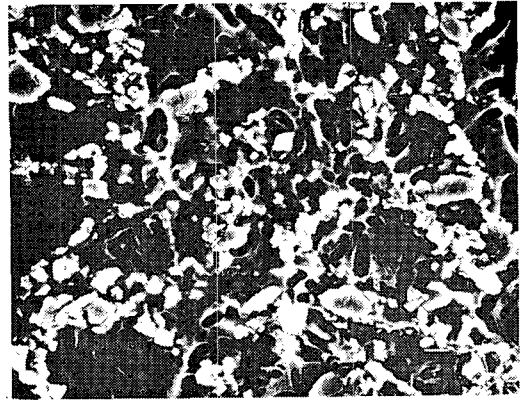
2.5 min. etch



5 min. etch



10 min. etch



20 min. etch

Fig. 6. SEM of porcelain sample at 500 magnification etched with a Excelco's porcelain etching gel



#### IV. DISCUSSION

The characteristics of dental porcelain using for aesthetic is low stress distribution, no firing deformation but easy to fracture in general. Many investigators<sup>29,38,47)</sup> studied the repair system of fractured PFM crown and porcelain jacket crown. Wide use of porcelain restoration caused necessity of porcelain repair systems. Retention of dental porcelain to composite resin was low and difficult to achieve. Jochen and Caputo<sup>30)</sup> recently reported on the effect of abrasive treatment on the temporary repair of porcelain denture teeth with a composite resin. They showed that any abrasive treatment substantially increased the retention of a composite build up. From a clinical point of view, it might be expected that the use of a coarse diamond would increase the retentive capacity provided by porcelain repair systems.

In 1955, Buonocore<sup>4)</sup> introduced the acid etch technique to promote adhesion of acrylic filling materials to enamel. Rochette<sup>49)</sup> was the first investigator to study the attachment of perforated cast metal frameworks to etched enamel via composite resin for splinting excessively mobile teeth. In 1979, Tanaka<sup>58)</sup> introduced a technique for inducing pitting corrosion on nickel – chromium – copper alloy which could be used for retention of resin to metal. Lividitis and Thompson<sup>59,60)</sup> demonstrated that a retentive surface for composite resin could be produced by electrolytically etching a nickel – chromium beryllium alloy.

Also acid etching technique was used for bonding porcelain to composite resin. Hydrofluoric acid was spreadly used in etching the porcelain surface. Etching porcelain with hydrofluoric based solution results in a myriad of microporosities with a honey comb – like appearance. Porcelain is a mixture of orthoclase

and potash; Feldspatic porcelain usually consists of 52% to 68% SiO<sub>2</sub>. The hydrofluoric acid will selectively degrade the SiO<sub>2</sub> forming microporosities on etched surface of the porcelain. ( $4\text{HF} + \text{SiO}_2 \longrightarrow \text{SiF}_4 + 2\text{H}_2\text{O}$  and  $\text{HF} + \text{SiF}_4 \longrightarrow \text{H}_2\text{SiF}_6$ ).

Hydrofluoric acid is well known agent for etching and polishing glass, which is an amorphous random network of SiO<sub>2</sub>.<sup>57)</sup> Calamia et al.<sup>7)</sup> showed that each porcelain had a specific concentrations and time of hydrofluoric acid etching for maximum bond. If the acid gel concentration was not known, or the time of application was inadequate, the result would be that of a less than optimum bonding surface. Horn<sup>23)</sup> reported porcelain etching method on porcelain laminate veneers bonded to etched enamel. Porcelain sample placed in a plastic beaker containing 10% hydrofluoric acid for 15 minutes in ultrasonic bath.

Recently, the use of hydrofluoric acid substitute increased due to easy manipulation. Calamia reported that porcelain samples were etched for 2.5, 5, 10, 20 min. with a hydrofluoric acid substitute, Stripit. The tensile bond strength obtained increased with the length of etching time. Hsu's<sup>25)</sup> study found that shear bond strength of resin to etched porcelain which was etched by Stripit for 2.5 minutes significantly improved the porcelain – resin bond using the silane coupling agent. Jones et al.<sup>31)</sup> showed that vidur N porcelain sample were acid – etched by means of varying concentrations and time of application. The best etched porcelain surface was produced by Stripit for 5 minutes in an ultrasonic bath. In my study, porcelain samples were etched by Excelco's porcelain etching gel and 5% HF solution for 2.5, 5, 10, 20 min. Both in the group etched with 5% HF solution and in the group etched with Excelco, the bond strength in relation to the lapse of etching time increased till 5 minutes, but had no great increase

after 5 minutes.

The study using hydrochloric acid or sulfuric acid as a porcelain etching solution was done by Calamia and Simonson. They concluded from his experiments that tensile bond strengths of composite resin to porcelain were significantly increased by etching of porcelain.<sup>53)</sup>

As expected, the etching of porcelain was the most significant factor in producing high bond strength. This could be noted from the Table 2. This clearly implicates micromechanical retention as a significant factor of the bond of composite resin to etched porcelain.

Silane coupling agent has been developed for chemical preparation of porcelain surface for bonding.<sup>2,9,17,37,39,40)</sup> Recently, hydrofluoric acid with silane coupling agent was introduced in porcelain laminate veneer technique.<sup>3,6,8,20,21,23,24,28,31)</sup> Bond strength of porcelain to composite resin obtained by using hydrofluoric acid with silane coupling agent was as follows: 2078 psi tensile strength by Calamia and Simonson<sup>6)</sup>, 3485 psi in shear strength by Hsu.<sup>25)</sup> Calamia and Simonson<sup>6)</sup> reported that best results were obtained using a combination of an etched porcelain with a specific silane coupling agent in bond strength.

The coupling agents are of the various silanes. Organosilane coupling agents are currently being used in some of the porcelain repair systems (Table 7).

Table 7. Organo-silane coupling agents

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r - aminophenyltriethoxysilane
vinyltriethoxysilane
r - Methacryloxypropyltrimethoxysilane

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Silane coupling agents, also known as adhesion promoters, function by absorbing onto and altering the surface of the solid (porcelain) to facilitate interaction by either chemical or physical process.<sup>17)</sup> The silane coupling agent will

react with silica (in porcelain dental crowns) to strong a chemical bond, once bonded to porcelain, is then free to bond chemically with any of the acrylic bonding materials. Not all dental porcelain crown or porcelain restorations are composition to which silane coupling agent will not bond.

Dentin/enamel bonding agents were developed to enhance seal and adhesion at both the dentin and enamel restorative interfaces. Enamel bonding agents are usually the unfilled Bis - GMA resin material. The rationale for using a resin of such low viscosities as a liner before inserting the composite itself is that good wetting of the resin to the tooth is assured by maximal penetration of the agent into the etched area in order to form the necessary resin tags. This procedure in intimate mechanical interlocking between the material and parts (organic and inorganic) of the enamel produces a stronger bond. Wettability is related to the intimate contact of liquid (the resin) in a solid (the microporosities of the etched enamel). Wettability is also a function of the viscosity (or rate of flow), which in turn is a result of the dilution of the highly viscous basic Bis - GMA resin.<sup>46)</sup>

But the composition of dentin does not allow the effective mechanical interlocking found in the enamel etching and bonding technique. Dentin is a nonhomogeneous structure composed of a large percentage of water and organic material. The composition makes it difficult to obtain a true chemical bond between dentin and resin restorations. Optimal results would be obtained by a strong chemical bond between the restorative material and the dentin. To achieve this object, dentin bonding agent has been developed.<sup>55)</sup> Majority of dentin bonding agent is one of multi-functional molecules possessing group intended to interact with the tooth surface and with the monomer in the composite.<sup>54)</sup> In the past few years, considerable

research has been devoted to determine the exact function of the bonding agent, since it was of a similar formulations as the composite resin matrix. The finding was divergent. Lucher and Otiz<sup>45)</sup> concluded that bonding agent was necessary to achieve proper bonding strength while still others did not think that the bonding agent was necessary at all.<sup>22,46)</sup>

In 1983, the 3M company introduced scotch bond, the first of series of commercially available products based on phosphonate ester systems. As in the Buonocore/Quigley approach, the phosphate groups played a key role by reacting with the calcium in the dentin and copolymerizing with the restorative resin. These unfilled resins are available in either chemically or photoactivated versions.<sup>16)</sup>

The use of silane coupling agent as an organo functional coupler between polymer and inorganic substances is well known. Although intended to work as a monolayer without adding film thickness, silane coupling agent more likely forms several monolayers which then condense and form oligomeric siloxanols. These can ultimately condense to crosslinked structures such as polymers. The further increase in bond strength in the unetched and etched porcelain group (resulting from the additional use of bonding agent) might be attributable in increased wetting as the interface, thus promoting condensation of silane to the composite. The bond strength of the group treated with silane coupling agent and bonding agent was highest.

The etched porcelain group selected demonstrated a microstructure that appeared most conducive to the development of high bond strength as a function of the number of large porosities contained of high bond strength as a function of the number of large porosities contained within its amorphous surface. Clearly, resin penetration of these spaces would enhance micromechanical bonding. The distinct differences in microstructure observed with each of

the concentrations of 5% HF solution and Excelco's porcelain etching gel suggested an associated preferential dissolution of one of the phases porcelain. In SEM analysis of porcelain etched surface, the group treated with 5% HF solution showed less etched pattern than with Excelco in relation to the lapse of etching time.

The current state of polymer technology allows resin – based dental restorative materials to be offered either as a chemically curing double – paste or as a light – curing single paste system. The latter group was divided by visible light composite resin and ultraviolet composite resin. In my study, the visible light composite resin was used, because a visible light composite resin has a some definite advantage as compared to ultraviolet curing system and chemically curing system.

The study did not test the effect of thermal cycling, water sorption, kinds of composite resin or bonding agent, concentrations of porcelain etching solution and type of porcelain etc. on the bond strength. These factors might affect each of these bonding systems of different degree in bonding strength. Therefore further study must be done about the these factors.

## V. CONCLUSION

The purpose of this study was to compare the effect of two etching solution, commercially available silane coupling agent and bonding agent, visible light composite resin on the tensile bond strengths of the etched porcelain at different etching time (2.5, 5, 10, 20 min.) and to observe the surface texture of the etched porcelain and unetched porcelain by using SEM.

There are three groups which are subdivided at three respectively. The specimens in group A were treated with 5% HF etching solution. In this group, a<sub>1</sub> was treated with silane coupling agent and visible light composite resin, a<sub>2</sub> was

treated with bonding agent and visible light composite resin and a<sub>3</sub> was treated with silane coupling agent, bonding agent and visible light composite resin at different etching time (2.5, 5, 10, 20 min.) The specimens in group B were prepared like those in group A, except group B with porcelain etching gel and control group with no etching.

Bond strength were determined after storage in 37°C water bath for 24 hours using a Shimadzu universal mechanical testing machine at a cross-head speed of 2 mm/min. The results were as follows.

1. Etched porcelain were higher than unetched porcelain in bond strength.
2. Bond strength of etched porcelain treated with silane coupling agent and bonding agent was highest and then, the group of treatment with silane coupling agent and the group of treatment with bonding agent in order.
3. Both in the group etched with 5% HF solution and in the group etched with Excelco, the bond strength in relation to the lapse of etching time increased by 5 min, but had no great increase after 5 min.
4. In differences of bond strength between the group etched with Excelco and the group etched with 5% HF solution, the group treated with bonding agent did not show statistical significance, ( $p > 0.5$ ) But the group treated with silane coupling agent and the group treated with silane coupling agent and bonding agent showed statistical significance. ( $p < 0.05$ ,  $p < 0.01$ ).
5. The group treated with Excelco than with 5% HF solution showed more etched pattern.

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## 국문요지

### 부식된 도재와 광중합성 수지와와의 접합강도에 관한 실험적연구

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#### 이 준 규 · 계 기 성

도재소부전장관에서 금속이 노출되지 않고 도재만이 파절시, 구강내에서 직접 행할 수 있는 심미적 수복 방법으로서 복합성 수지를 이용한 수복 방법이 널리 이용되고 있는데 본실험의 목적은 도재와 광중합성 수지간의 접합강도를 비교 측정코자 함에 있다.

본 실험에서는 주사전자 현미경을 사용하여 도재의 부식액 (5% 불화수소산용액, Excelco's porcelain etching gel) 및 부식 시간 (2.5분, 5분, 10분, 20분) 에 따른 부식양상을 먼저 관찰하였다. 그다음 각기 다른 시간에서 부식 처리된 도재와 대조군으로서 미부식 처리된 도재에 광중합성 수지를 부착시킬시, 첫째 silane coupling agent (Scotch-Prime) 도포후 광중합성 수지를 결합 시켰고 둘째 bonding agent (Scotch bond) 도포후 광중합성 수지를 결합시켰으며 셋째 Silane coupling agent (Scotch-Prime) 에 bonding agent (Scotch bond) 를 도포후 광중합성 수지로 결합 시킨후, 인장강도 측정기 (Shimadzu universal testing machine) 를 사용하여 결합력을 측정 한 결과 다음과 같은 결론을 얻었다.

1. 부식처리된 도재가 미부식 처리된 도재 보다 결합력이 높았다. ( $p < 0.05$ ,  $P < 0.001$ )
2. 부식 처리된 도재나 미부식 처리된 도재에 있어서 silane coupling agent 도포 후 bonding agent 로 처치한 경우 가장 결합력이 높았으며 그다음 silane coupling agent 로 처치한 경우, bonding agent 로 처치한 경우의 순이었다.
3. 도재부식 시간의 증가에 따른 결합력의 차이는 5% HF 용액으로 부식 처리된 도재와 Excelco 로 부식 처리된 도재에 있어서 5분 정도 까지는 결합력의 증가를 보였으나 그 후에는 결합력에 있어서 거의 증가하지 않는 양상을 보였다.
4. Excelco 로 부식 처리된 도재와 5% HF 용액으로 부식처리된 도재간의 결합력 차이에 있어서 bonding agent 만으로 처치한 경우에는 두결합력 사이에 서로 유의한 차이를 보이지 않았으나 ( $P > 0.5$ ) silane coupling agent 만으로 처치한 경우와 silane coupling agent 도포후 bonding agent 로 처치한 경우에는 두결합력 사이에 서로 유의한 차이를 보였다. ( $P < 0.05$ ,  $P < 0.001$ )
5. Excelco 로 부식처리된 도재가 5% HF 용액으로 부식처리된 도재보다 부식정도가 더 현저하였다.