CHAPTER III

MATERIALS AND METHODS

The material and methods is divided into three parts as follows:

3.1 MATERIALS AND INSTRUMENTS

3.2 THE EXPERIMENTAL PROCESS

3.2 STATISTICAL ANALYSES

3.1 MATERIALS AND INSTRUMENTS

3.1.1 Sample

The samples in this study were feldspathic porcelain disks (Figure 3.1) which were fabricated from conventional feldspathic porcelain (Vita porcelain powders, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Bad Säkingen, Germany). All the specimens tested in this study were made from the same rubber mold. Porcelain disks, 10 mm in diameter and 5 mm in thickness, were prepared according to the manufacturer's recommendations by skilled ceramic technician.

Figure 3.1 Porcelain specimens

3.1.2 Brackets

All brackets used in this study were 0.018" X 0.025" slot, Mini Diamond type, metal standard edgewise upper central incisor brackets (Ormco Corporation, Orange, California, USA). The total area of each bracket was 0.1064 cm^2.

Figure 3.2 Metal standard edgewise upper central incisor bracket

3.1.3 Materials

Etching

- Conventional 37% phosphoric acid (Etching Solution, Ormco Corporation)
- 9.6 % hydrofluoric acid (HF) (Porcelain etch gel, PULPDENT Corporation, Watertown, Massachusetts, USA)

Silane

- Silane agent (Porcelain liner M, Sun Medical Co., Ltd., Shiga, Japan)

Adhesive resin

- Self-cured adhesive resin (System™1+, Ormco Corporation)
- Resin cement (Super-Bond C&B, Sun Medical Co., Ltd.)

Figure 3.3 Phosphoric acid (Etching Solution, 37% phosphoric acid)

Figure 3.4 Hydrofluoric acid (Porcelain etch gel, 9.6 % hydrofluoric acid)

Figure 3.5 Silane agent - (Porcelain liner M)

Figure 3.6 Self-cured adhesive resin (System™1+**)**

Figure 3.7 Resin cement (Super-Bond C&B)

3.1.4 Instruments

1. An Instron® 5566 universal testing machine (Instron Calibration Laboratory, Norwood, Massachusetts, USA) was used for measuring shear bond strength value (Figure 3.8)

Figure 3.8 Universal testing machines (Instron® 5566)

2. A de-bonding plate and a mounting jig (Figure 3.9)

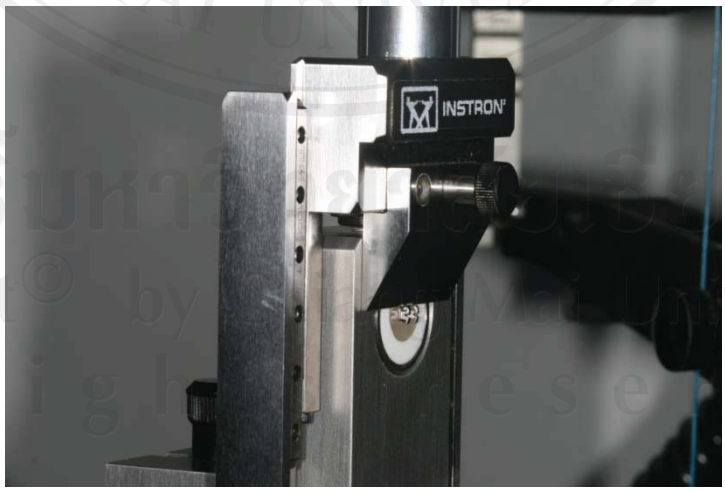

Figure 3.9 De-bonding plate and a mounting jig

3. A Memmert Model 200 incubator (Memmert GmbH + Co. KG, Schwabach, Germany) was maintained at 37°C for this study. (Figure 3.10)

Figure 3.10 Incubator (Memmert Model 200)

4. BioSonic® Model UC 125 Ultrasonic cleaner (Coltène/whaledent AG, Altstätten, Switzerland) for cleaning specimen surfaces (Figure 3.11)

Figure 3.11 Ultrasonic cleaner (BioSonic® Model UC 125)

5. Model HWB332R, TC 301, CWB332R Model HWB332R, TC 301, CWB332R Thremocycling device (Medical & Environment Equipment Research

Laboratory King Mongkut's Institute of Technology Ladkrabang., Thailand) (Figure 3.12)

Figure 3.12 Thermocycling device

3.2 THE EXPERIMENTAL PROCESS

The experimental process was divided into five parts as follows:

3.2.1 Specimen preparation

3.2.2 Direct bonding procedure

3.3.3 Storage and thermocycling

3.3.4 Shear bond strength testing

3.4.5 Examination of the failure modes

3.2.1 Specimen preparation

The surfaces of the porcelain disks were exposed and positioned parallel to the rims of the rings (Figure 3.13) on flat surface. The self-curing resin was pouring in to the rings to fix the specimens. The porcelain surfaces that will be bonded with orthodontic brackets were exposed parallel to the rim of the rings in order to obtain

stability during the bond strength tests. The parallelism (Figure 3.14) enabled a standardized force direction to the bracket when testing. The surfaces of the specimens were then finished with #480, 600, 1000 and #2000 waterproof abrasive papers, under running water. The samples were then placed in an ultrasonic cleaner for 10 minutes to clean and remove debris.

Figure 3.13 The surface of the porcelain disk was exposed parallel to the rim of the ring (bottom surface)

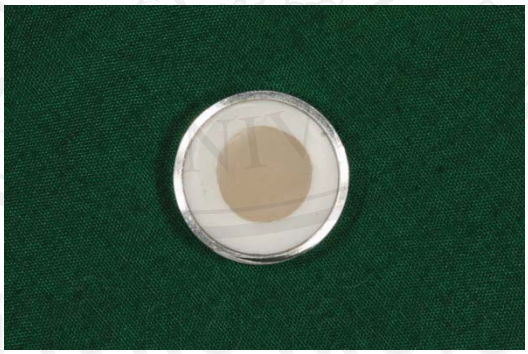

Figure 3.14 Porcelain specimen - embedded disk with self-curing resin in stainless steel ring

3.2.2 Direct bonding procedure

The porcelain specimens were randomly distributed to five test groups (Table 3.1). Each group had 20 specimens.

Table 3.1 Five groups of adhesive systems used in this study

Systems	Acid etching	Silane	Adhesive
Group I	Porcelain etch gel (9.6% Hydrofluoric acid)	-	System™1+
Group II	Etching Solution (37% phosphoric acid)	Silane	System™1+
Group III	Porcelain etch gel (9.6% Hydrofluoric acid)	Silane	System™1+
Group IV	Etching Solution (37% phosphoric acid)	Silane	Super-Bond C&B
Group V	Porcelain etch gel (9.6% Hydrofluoric acid)	Silane	Super-Bond C&B

The orthodontic brackets were bonded to the specimens according to one of five protocols:

Group I: (9.6% Hydrofluoric acid + System™1+)

The specimen surfaces were etched with Porcelain etch gel (9.6% hydrofluoric acid) for 60 seconds, washed and dried. The brackets were then placed on the specimens, using System1™+, according to the manufacturer's instructions. The liquid activator was applied on the etched porcelain surfaces and the bracket bases. Then, the composite resin was applied to the bracket bases. After the brackets were firmly placed on the porcelain surfaces, excessive resin was removed with an explorer.

Group II: (37% Phosphoric acid + Silane + System™1+)

The specimens were etched with Etching Solution for 60 seconds, followed by rinsing and drying. One coat of porcelain liner M was applied to the porcelain surfaces. The brackets were then placed on the specimens, using System™1+, according to the manufacturer's instructions.

Group III: (9.6% Hydrofluoric acid + Silane + System™1+)

The specimen surfaces were etched with Porcelain etch gel (9.6% hydrofluoric acid) for 60 seconds, washed and dried and one coat of porcelain liner M was then applied. The brackets were then placed on the specimens, using System™1+, according to the manufacturer's instructions.

Group IV: (37% Phosphoric acid + Silane + Super-Bond C&B)

Etching Solution was applied for 60 seconds, followed by rinsing and drying. One coat of porcelain liner M was applied to the porcelain surfaces. The brackets were then bonded using Super-Bond C&B. The catalyst, partly oxidized tri-n-butylborane (TBB), was added to the monomer liquid to prepare an activated monomer liquid. The polymer powder and activated monomer liquid were mixed and used to bond the metal brackets to the porcelain surfaces using the brush-dip technique. Excess adhesive was removed with an explorer.

Group V: (9.6% Hydrofluoric acid + Silane + Super-Bond C&B)

The specimen surfaces were etched with Porcelain etch gel for 60 seconds, washed and dried. One coat of porcelain liner M was applied to the porcelain surface. The brackets were bonded using Super-Bond C&B according to the manufacturer's instructions, using the brush-dip technique as in Group IV.

After bonding, the specimens were left undisturbed for one hour.

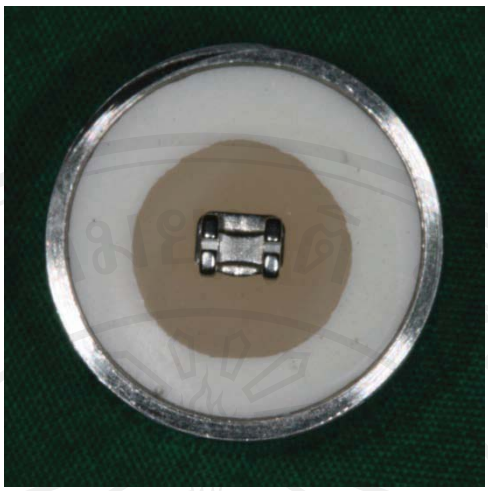

Figure 3.15 Porcelain specimen with bonded bracket

3.2.3 Storage and thermocycling

After the adhesive resin was completely set, the specimens were stored in distilled water at 37ºC for 24 hours. The samples were then thermocycled for 1000 cycles with temperature limits set at 5±2°C and 55±2°C, with an immersion time of 30 seconds in each distilled water bath, and traveling between the two water baths in room temperature air for 10 seconds.

3.2.4 Shear bond strength testing

Each specimen with bonded brackets was mounted into the jig, which was fixed into the lower pneumatic grip (Figure 3.16). Shear bond strength values were determined by using a universal testing machine (Instron® 5566) with a five-kilonewton load cell, at a 0.5 millimeter per minute crosshead speed. The values of shear bond strength at bond failure were recorded.

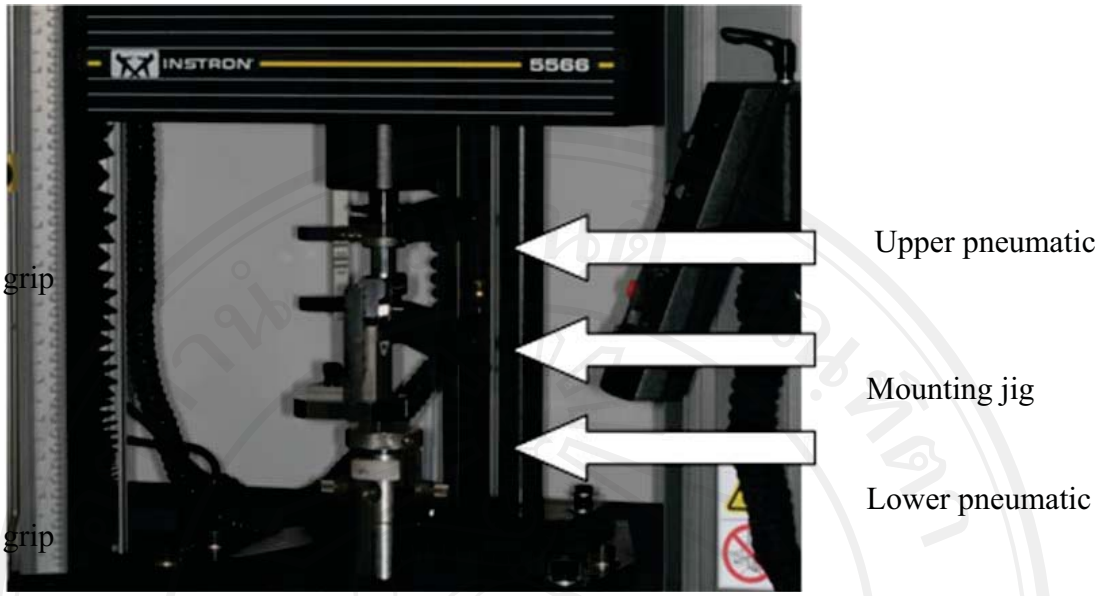

Figure 3.16 The mounting jig was fixed into the lower pneumatic grip.

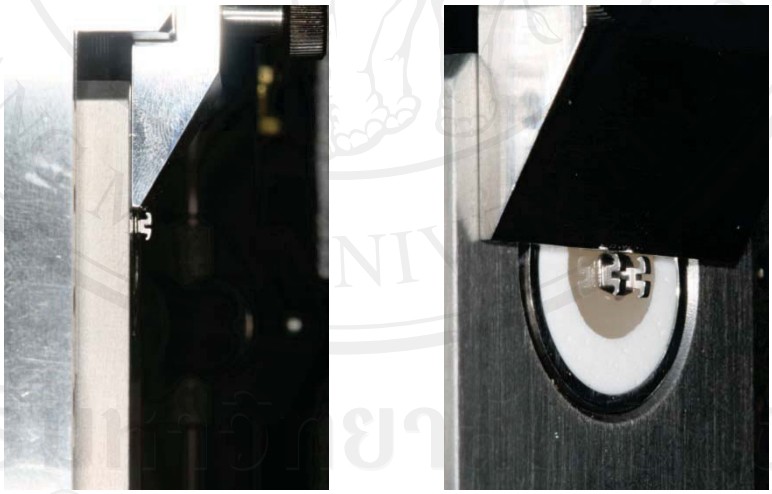

Figure 3.17 The apparatus assembled for testing shear bond strength

3.2.5 Examination of the failure modes

After de-bonding, failure sites were determined by examination of the de-bonded bracket surfaces. The amounts of residual adhesives on the de-bonded

bracket surfaces were determined from pictures (Figure 3.18) that scanned with 1200 dpi from scanner (HP laser jet 3390). The computerized transparent grid was placed on the pictures (Figure 3.19). The percentages of residual adhesives on the bracket surfaces were converted to residual adhesive per total de-bonded porcelain surface. Visual inspections of all de-bonded porcelain surfaces were carried out and the specimens with visible porcelain surface damaged were assigned for score 4.

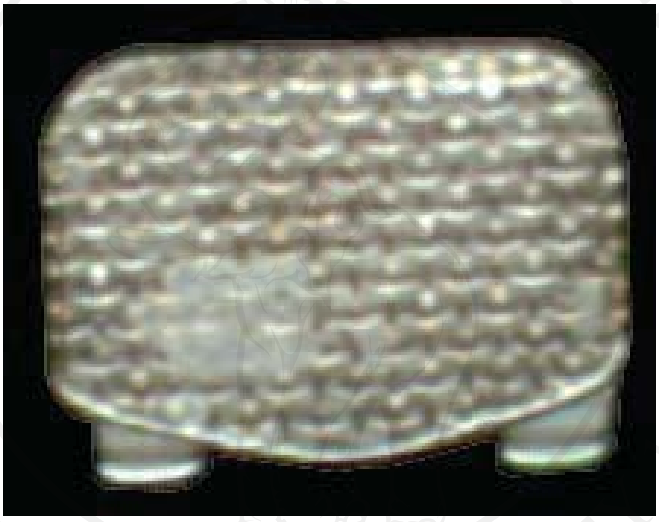

Figure 3.18 Scanned picture of de-bonded bracket surface

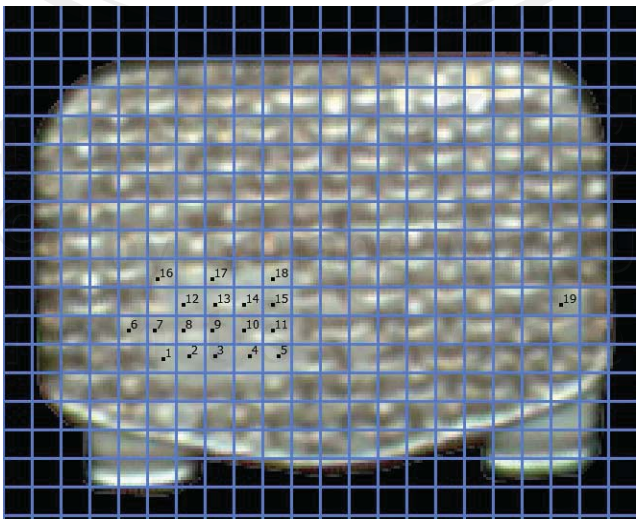

Figure 3.19 De-bonded bracket surface with computerized transparent grid

A modified Adhesive Remnant Index (ARI) was used to evaluate the amount of adhesive left on the porcelain sample and to evaluate damage to the porcelain after de-bonding.[37] The index was modified from the Adhesive Remnant Index of Artun and Bergland, 1984[37] by including a score of 4 for samples with damaged porcelain surfaces.[13,14]

Modified Adhesive Remnant Index (ARI):

Score 0 =No adhesive left on the porcelain surface (adhesive/porcelain interface failure)

Score 1 = Less than half the adhesive left on the porcelain surface

Score 2 = More than half the adhesive left on the porcelain surface

Score 3 = All the adhesive left on the porcelain surface, with a distinct impression of the bracket mesh (adhesive/bracket interface failure)

Score 4 = Damage to the porcelain sample

3.3 STATISTICAL ANALYSES

Windows SPSS version 14.0 was used to calculate the following analyses:

1. The values of shear bond strength of five different adhesive systems were described using means, standard deviations values.
2. One way analysis of variance (ANOVA) was used to compare the mean shear bond strength values among five different adhesive systems.
3. A multiple comparisons test (Tukey's test) was used to identify which groups were different when there was a significant difference in the mean shear bond strength value among five different adhesive systems.
4. Descriptive statistic was used to analyze the ARI scores.