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석사학위 논문

자가중합레진으로 부착된 금합금과 금속 브라켓의 결합강도에 silica coating이 미치는 영향

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치의학과

류 민 주

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Effect of silica coating on bond strength between a gold alloy and metal bracket bonded with chemically cured resin

2014년 8월 25일

조 선 대 학 교 대 학 원

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이 논문을 치의학 석사학위신청 논문으로 제출함

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ABSTRACT

자가중합레진으로 부착된 금합금과 금속 브라켓의 결합강도에 silica coating이 미치는 영향

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Objective: 본 연구는 자가중합레진을 사용하여 금속 브라켓을 금합금에 부착시에 세 가지 다른 금합금 표면처리방법의 효과를 비교하고자 하였다.

Methods: 210개의 type III 금합금 시편들이 세 가지 다른 표면처리방법(aluminum oxide sandblasting/aluminum oxide sandblasting 후 metal primer 도포/silica coating과 silanation)과 열순환 처리 유무에 따라 6개 군으로 무작위 분류되었다. 시편들을 각 조건에 맞게 표면 처리한 후에, 자가중합레진(Unite®, 3M Unitek, Monrovia, CA, USA)으로 금속 브라켓을 금합금 시편 표면에 부착하였다. 금합금 표면에서 브라켓이 탈락되는 순간에 전단결합강도(SBS)가 측정되었고, 시편 표면에 남아있는 레진이 Adhesive remnant index(ARI)로 평가되었다.

Results: 금합금 표면에 Aluminum oxide sandblasting 처리 후 metal primer를 도포한 군이 aluminum oxide sandblasting만을 시행한 군보다 더 높은 결합강도를 보였으며($p < 0.001$), silica coating과 silanation 처리한 군이 aluminum oxide sandblasting 후 metal primer를 처리한 군보다 더 높은 결합강도를 나타냈다($p < 0.001$). 열순환 처리를 시행한 모든 군에서는 전단결합강도에 있어 유의한 변화를 보이지 않았다.

Conclusion: 자가중합레진을 사용하여 금속 브라켓을 금합금에 부착 시, 금합금 표면에 silica coating과 silanation 처리를 하면 임상적으로 만족할 만한 결합강도를 얻을 수 있을 것으로 사료된다.

I. Introduction

Orthodontic treatment for middle-aged patients is increasing, and middle-aged patients typically exhibit more dental restorations and a poorer periodontal status compared to adolescents and young adults. Orthodontic bands are typically used for restored teeth where direct bonding of the bracket is difficult. However, dental plaque may accumulate around the band margins[1] as a result of the difficulty of fitting the bands to tooth surfaces. Although surface conditioning techniques and adhesion materials for direct bracket bonding to the metal surface have been developed, the bond strength of the adhesives to the gold alloy is less satisfactory than classical method using acid etching of enamel surface.

Adhesion is defined as "the maintained status of two materials at the interface by mechanical and chemical bonding forces." Acid etching of enamel is a good example of enhancing mechanical bond strength. As fine irregularities are formed at the enamel surface by acid etching, efficient and strong adhesion can be achieved by the micromechanical lock effect as bonding agents with desirable flowability are infiltrated into fine irregularities.[2] However, the metal surface cannot be acid-etched like enamel. Therefore, macromechanical retention with green stone[3] and diamond bur, and micromechanical retention with aluminum oxide sandblasting[4] are widely used in clinical situations. In addition to the methods used for enhanced mechanical retention, chemical bonding between the metal surface and the adhesion materials using a metal primer has been studied.[5,6] The bond strength of these methods may not reach to the bond strength of enamel etching, and there is a controversy over the sustainability of the enhanced bond strength obtained by the application of metal primer.[6] However, more and more orthodontists tend to use the direct bonding to the metal restorations because of the advantage in the aspect of oral hygiene, as well as convenience to the practitioner, compared to the conventional method using bands. Recently, chemicophysical bonding with a silica coating and silanation (silicoating)[7,8], which can enhance the mechanical and chemical bonds, has been introduced in the field

of orthodontics. Chemically cured resin can be more fully polymerized than the light cured resin especially in the central area of the bracket when a metal bracket is bonded to the gold alloy.[9] This suggests that the chemically cured resin could be better in this circumstance. Metal primer is popular among orthodontists because it can be applied easily after routine sandblasting using aluminum oxide. Until now, the bond strength attained by different surface conditioning methods including sandblasting, application of metal primer, and silicoating was not thoroughly compared in the condition of using chemically cured resin for orthodontic bonding system.

The purpose of this study was to compare the shear bond strength (SBS) of three surface conditioning methods including aluminum oxide sandblasting only, aluminum oxide sandblasting and application of metal primer, and silicoating when a metal bracket is bonded to a gold alloy surface using chemically cured resin. The change in the SBS after thermocycling was also investigated in order to examine the influence of the stress caused by repetitive temperature changes, which is the primary cause of bond strength weakening in clinical practice. The adhesive remnant index (ARI)[10] was also evaluated.

II. Materials and methods

Fabrication of experimental specimens

Plate-shaped specimens of type III dental gold alloys used for casting (Goldenian C-48, Shinhung, Seoul, Korea) were placed at the bottom of a cuboidal silicon mold (2.4 cm x 2.4 cm x 1.9 cm). Because the type III dental gold alloy is primarily used for onlay, crown, and crown and bridge in small areas, the type III gold alloy is thought to be appropriate for specimens in this investigation, assuming that the adherend is a posterior tooth restoration. The gold alloy used in the present study contains 48% gold, 38.2% silver, 4.0% Pd, and 9.8% microelement. The orthodontic acrylic resin (Dentsply, York, PA, USA) was poured into the silicon mold. The specimen was exposed at the top of the block when the mold was removed after the resin blocks were completely cured.

The gold alloy surfaces were sequentially polished with No. 120, 1000, 2000, and 3000 sandpapers (Single disc polisher, Gang Hae Industry, Gwangju, Korea) under a spray of water, and then the specimens were high polished with a rouge for dental alloys (Hi-rouge®, High Dental Korea, Seoul, Korea) and a cotton wheel (Dae-Myung Dental, Seoul, Korea) in order to bond the brackets to flat surfaces of the gold alloys. The adhesion area was limited to a size of 5 mm × 5 mm by applying a scotch tape to the final polished gold alloy surface, with the exception of the adhesion area. The total number of gold alloy specimens for this experiment was 210, and the specimens were randomly grouped into 70 samples to be treated with one of the three surface conditioning methods. Half of each group was reclassified for thermocycling. Finally, the gold alloy specimens were classified into six groups, consisting of 35 samples per group (Table 1).

Bracket bonding

The specimen surfaces of the AS, AST, MP, and MPT groups were roughened by an intraoral sandblaster (Microetcher, Danville materials, San Ramon, CA, USA) filled with 50 μm-sized aluminum oxide (Blasting medium, Dentaurum,

Ispringen, Germany) at a distance of 1 cm, with 3.6 kgf/㎠ of pressure for 10 s. Afterward, the scotch tape was removed from the surfaces of the specimens, and the surfaces were rinsed and dried thoroughly with an air-water spray for 10 s each. A uniform coat of metal primer (Metal primer, Reliance orthodontic products, Itasca, IL, USA) was applied onto the gold alloy specimens of MP and MPT groups with a microbrush. The specimens were then allowed to be dried for 30 s according to the manufacturer's instructions.

30 μm silicon dioxide (Cojet-Sand, 3M ESPE, Seefeld, Germany) was sandblasted with an intraoral sandblaster (Microetcher, Danville materials, San Ramon, CA, USA) at a distance of 1 cm with 3.6 kgf/㎠ of pressure for 10 s for the SC and SCT groups. The gold alloy surfaces of these groups became roughened and silica-coated. Afterward, the scotch tape was removed from specimens and dry air was lightly applied for 5 s. Silane (ESPE-Sil, 3M ESPE Dental Products, St. Paul, MN, USA) agent was applied with a microbrush and evaporated for 5 min according to the manufacturer's instructions.

Standard metal brackets for the upper central incisor (Standard edgewise bracket, TOMY, Tokyo, Japan) with a base area of about 14.22 mm^2 were bonded to all specimens that underwent surface conditioning. The bracket bases were not specifically conditioned prior to bracket bonding. The bonding adhesives used in this study was a chemically cured resin (Unite®, 3M Unitek, Monrovia, CA, USA). The bracket base area and the gold alloy surfaces were coated with a resin primer. And a resin adhesive paste was then applied to the bracket bases in small amounts, according to the manufacturer's instructions. The brackets were subsequently positioned and bonded to the gold alloy surfaces with constant pressure, and the excess resin extruded at the bracket-alloy interface was carefully removed with an explorer. Specimens were kept at room temperature for 4 min, allowing chemical curing to take place. After this process, they were stored in a 36.5°C water bath (Water bath, Jeio Tech., Daejeon, Korea) with 100% relative humidity for 24 h in order to achieve a complete cure. A series of processes such as the fabrication of gold alloy blocks, surface conditioning, and bracket bonding were carried out by the same researcher for consistency and

reproducibility of the study.

Thermocycling

The AST, MPT, and SCT groups were stored in the water bath for 24 h and subsequently thermocycled, based on the work of Bishara et al.[11] Specimens were thermocycled 1000 times in 5°C and 55°C distilled water baths (Thermocycler, Gang Hae Industry, Gwangju, Korea). The samples were immersed in each bath for 20 s, which was considered to be one cycle of thermocycling.

Measurement of SBS

A universal testing machine with 500 N load cell (Lloyd Instruments®, Ametek, Berwyn, PA, USA) was used to measure the SBS of each specimen. The acrylic resin blocks were fixed into the lower jig vice. The anterior/posterior and right/left positions of the acrylic resin blocks were adjusted to make the adhesive face between the bracket and the specimen be perpendicular to the crosshead (Figure 1). The crosshead speed for the SBS measurement was established as 1 mm/min, and the maximum load at the moment of bracket debonding was measured. The SBS (MPa) of each specimen was measured by dividing this numerical value (N) with the bracket's base area (mm^2).

ARI measurement

Each adhesive face of the gold alloy specimens was photographed with a digital camera (Nikon D90, 105 mm micro lens, 1/125, F25, ISO 200, -0.3 eV) after measuring the SBS. All photographed images were cropped, except for the adhesive face area on the image processing software (Photoshop CS5 Extended 12.0, Adobe systems Inc., San Jose, CA, USA). Grids with 1 mm width and length were drawn in the cropped images, and the resin remnants were evaluated using the ARI of the edited images on a monitor. The criteria for evaluating the ARI[10] are shown in Table 2.

Statistical analysis

The Shapiro-Wilk test was performed and all six groups followed a normal distribution. Two-way analysis of variance (ANOVA) was used to assess the effect of the surface conditioning methods and thermocycling process on the SBS. The Kruskal-Wallis H test was used to investigate the statistical significance of the ARI among groups for the different surface conditioning methods, and the Mann-Whitney U test was performed with Bonferroni correction for multiple comparisons. The Mann-Whitney U test was used to evaluate the effect of thermocycling on the ARI. And the correlation between the SBS and ARI for each group was evaluated by the Spearman correlation analysis. All statistical anaylses were performed using SPSS 12.0 (SPSS, Chicago, IL, USA).

III. Results

The mean value, and standard deviation of the SBS for each group, and the statistical significance of the SBS among the groups are described in Table 3. The mean SBS of the AS, MP, and SC groups were 14.0, 18.7, and 21.9 MPa, respectively. The MP group exhibited a higher SBS value than the AS group ($p < 0.001$), and the SC group showed a higher SBS value than the MP group ($p < 0.001$) (Table 3).

Although the mean SBS of the MPT and SCT groups slightly decreased after thermocycling, no significant difference of SBS was observed after thermocycling ($p = 0.133$) (Table 3). And there was no interaction between the surface conditioning methods and thermocycling process ($p = 0.340$).

The distribution of the ARI for each specimen is shown below (Table 4). A score of 0 in the ARI was most frequently observed in the AS and AST groups; a score of 1 was most frequently observed in the MP and MPT groups; and a score of 2 was most frequently observed in the SC and SCT groups (Table 4, Figure 2, Figure 3, Figure 4).

The results of the Kruskal-Wallis H test and the Mann-Whitney U test with Bonferroni correction ($p < 0.0167$) revealed the significant differences of ARI among three surface conditioning methods regardless of thermocycling process. The Mann-Whitney U test showed that there was no statistical difference of the ARI between the AS and AST groups ($p = 1.0$), or between the SC and SCT groups ($p = 0.194$) (Table 4).

The SC and SCT groups exhibited a higher SBS value than the MP and MPT groups, respectively. The MP and MPT groups showed a higher SBS value than the AS and AST groups, respectively (Table 3). Similar results are shown in the ARI (Table 4). Based on these results, the correlation between the SBS and ARI was investigated, and a moderately positive correlation was observed between the SBS and ARI (Spearman correlation coefficient = 0.637, $p < 0.05$).

IV. Discussion

Several studies[7,12,13] reported that the aluminum oxide sandblasting effectively enhance the micromechanical retention between the metal and the adhesive resin. In the present study, there was no significant change in the bond strength after thermocycling, similar to the result of previous study.[13]

The effect of chemical conditioning methods using metal primers on the bond strength also have been evaluated.[6,8,14] Among the various commercialized metal primers, it is known that the acid anhydride group of the metal primer containing 4-META (methacryloxyethyl trimellitic anhydride) combines with the hydroxyl groups and oxygen atoms in the metal oxide layer, and methacrylate group of 4-META polymerizes with the composite resin.[8,12] By this mechanism, the metal primer containing 4-META increases the bond strength between the metals and the composite resin.[8,12]

In the present study, sufficient bond strength was maintained after thermocycling when the metal primer was used after sandblasting. This result corresponds with the investigation by Matsumura et al.[15] in which the samples were thermocycled after V-primer and Metalite application. However, Lee et al.[6] found that metal primers including V-primer, Metaltite, and the Alloy primer significantly increased the bond strength to the gold alloys, while the bond strength of the specimens treated with all three types of primer decreased after thermocycling, and concluded that the metal primer had no advantage in the bond strength over aluminum oxide sandblasting. This result is contrary to the result of the present study, and the reason seems to be the use of different resin types in each study. Yoshida and Atsuta[16] reported that the effectiveness of the primers for noble metals may be influenced by the initiator system of the resin bonded to the metal. According to their study, when V-primer was applied to gold alloys, the tri-n-butylborane (TBB) resin and benzoyl peroxide-amine resin exhibited a higher bond strength to gold alloys relative to the camphoroquinone-amine resin, with or without thermocycling.[16] Lee et al.[6] used a light cured resin containing

camphoroquinone as the photoinitiator. There has not yet been a study that evaluates whether the bond strength of the gold alloy specimens treated with the metal primer containing 4-META is maintained after thermocycling. Therefore, no data is available for direct comparison to our study.

If abrasive particles with a silicated surface are blasted onto the metal surface at high energies, then the high temperature formed by the impact of abrasive particles produces silica to be incorporated and embedded into the metal to a depth of 15 ㎛, converting kinetic energy into thermal energy.[17] Atsü et al.[18] reported that the silica particles were incorporated into the metal surface by blasting pressure, forming a fine roughened surface when the metal surface was blasted with 30 ㎛-sized aluminum oxide, chemically modified with silica (Cojet-Sand, 3M ESPE, Seefeld, Germany). Watanabe et al.[19] verified that a silica layer was formed on the surface of dental gold alloys using electron microprobe analysis element mapping.

When the silane is applied to the silica layer formed on the metal surface, silane and silica are known to strongly combine by a dehydration reaction, separating the hydroxyl group.[8] Silanes have a dual reactivity. Non-hydrolyzable functional groups can combine with the resin composite monomers containing C=C double bonds, and the hydrolyzable alkoxy group can bond with the hydroxyl group rich inorganic substrates such as silica.[18,20] Because silane supplied as a conditioner is a liquid dissolved in a volatile solvent such as alcohol, its application is not difficult. However, some time for the evaporation of the solvent is required for a stronger bond, and the chemical reaction between silica and silane also requires time. Therefore, an appropriate time interval would be necessary for successful silicoating procedures.[21] Manufacturers recommend 5 min of silanation before applying the adhesive for the extraoral use, and 1 min for the intraoral use. In this study, a time interval of 5 min after silane application was chosen to avoid the effect of insufficient time for the evaporation of the solvent and chemical reaction. In clinical applications, 1 min after silane application may be adequate.

As the silicoating is achieved by incorporation of silica particles into the metal surface, this method is less affected by the gold alloy composition and oxide layer

formation, compared to the metal primer application after aluminum oxide sandblasting.[8,22] In the present study, silicoating group exhibited SBS of 20.73 ± 2.46 (mean ± SD) MPa after thermocycling, which is higher bond strength than the metal primer application after aluminum oxide sandblasting. This SBS value is comparable to the SBS of metal bracket bonded to the enamel surface.[23] Also, This SBS value is stronger than the SBS value of 13.63 ± 2.55 MPa reported in the study of Jung et al.[8] in which light cured resin was used with silicoating.

Typically, the weaker the bond between the adherend and the adhesives, the lesser the resin remnants will be left on the adherend surface[21,24]; this trend was also observed in the present study. More resin remnants were left on the gold alloy specimens in the SC group than the MP group, and more remnants were present in the MP group than the AS group. This trend was maintained after thermocycling. According to a study by Jung et al.[8], the ARI was higher when the silicoating was used instead of aluminum oxide sandblasting; however, there was no statistical significance. Jung et al.[8] regarded the cause of the result as the use of light cured resin adhesives in their study, and reported that the insignificant difference in ARI might have been produced by uncured resin remaining nonuniformly on the specimen surface.

Although the bond strength would be increased when treated with a metal primer after aluminum oxide sandblasting, more satisfactory bond strengths can be attained when the silicoating is used. Based on this result, the success rate of direct bonding to the gold alloy restorations or prosthesis can be increased by the silicoating to the surfaces of the gold alloys.

Silicoating may increase the bond strength in both light cured resin[8] and chemically cured resin. But there may be difference in the bond strength and ARI according to the resin types. This should be studied in the future. And the brackets were bonded to flat surface of the gold alloys in the present study, but the actual surface of restorations with gold alloys is not flat. So the gap between the surface of gold alloys and brackets can be formed inevitably and the clinical bond strength would be less than the value of the in-vitro study when chemically cured resin is used. Clinical studies are required regarding to the bond failure rate.

V. Conclusion

When a chemically cured resin is used to bond metal brackets to gold alloys using surface conditioning methods of aluminum oxide sandblasting alone, metal primer application after aluminum oxide sandblasting, or silicoating,

1. The highest SBS can be attained with silicoating technique.
2. The metal primer application after sandblasting increases SBS compared to sandblasting alone.
3. The SBS is maintained after thermocycling for all surface conditioning methods.

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Tables

Table 1. Experimental groups classified by surface conditioning method and thermocycling.

Surface conditioning	Thermocycling	
	No	Yes
Al_2O_3 sandblasting	AS	AST
Al_2O_3 sandblasting + metal primer	MP	MPT
Silica coating + silane	SC	SCT

Table 2. The criteria of evaluation of ARI

ARI	Criteria
0	No adhesive left on the specimen
1	Less than half of the adhesive left on the specimen
2	More than half of the adhesive left on the specimen
3	All adhesive left on the specimen

ARI, Adhesive remnant index.

Table 3. The mean value and standard deviation of shear bond strength of samples and statistical significance of shear bond strength between groups.

Group	Shear bond strength (MPa)	Sub group	Significance
AS	14.00 ± 3.28	a	Surface conditioning* AS < MP < SC
AST	14.21 ± 2.99	a	
MP	18.72 ± 3.41	b	
MPT	17.82 ± 2.49	b	Thermocycling AS=AST(p=0.133), MP=MPT(p=0.133) SC=SCT(p=0.133)
SC	21.94 ± 3.34	c	
SCT	20.73 ± 2.46	c	

The shear bond strength value is presented as mean ± standard deviation.
Measurements with the same subgroup letter indicate no statistically significant difference using multiple comparisons with Tukey's test at a significance level of $p > 0.05$.

* indicates a significant result after two-way analysis of variance.

Table 4. Each experimental group's ARI and statistical significance of ARI between groups

Group	ARI					Sub group	Significance
	0	1	2	3	N		
AS	35	0	0	0	35	a	Surface conditioning* AS < MP < SC
AST	35	0	0	0	35	a	
MP	1	25	9	0	35	b	
MPT	9	26	0	0	35	c	Thermocycling MP > MPT* AS=AST (p=1.000),SC=SCT(p=0.194)
SC	0	5	28	2	35	d	
SCT	2	9	21	3	35	d	

ARI, Adhesive remnant index.
Measurements with the same subgroup letter indicate no statistically significant difference using Mann Whitney U-test at a significance level of $p > 0.05$.

* indicates a significant result after Mann Whitney U-test.

Figures

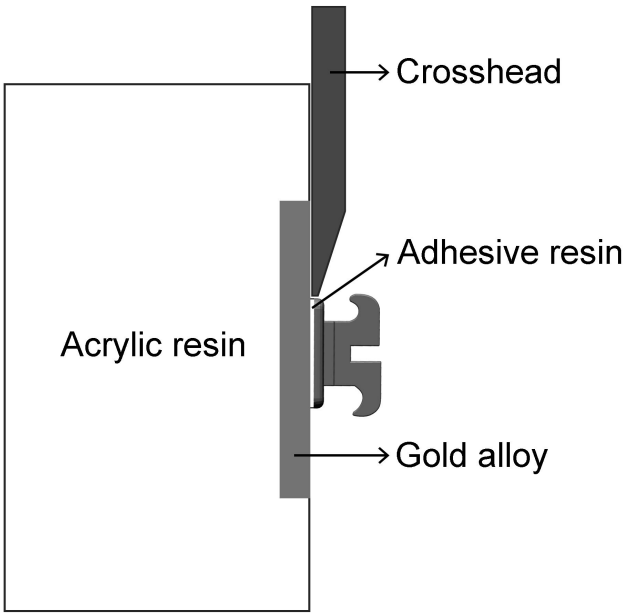

Figure 1. Schematic diagram of specimen used in experiment, and crosshead position for shear bond strength measurement.

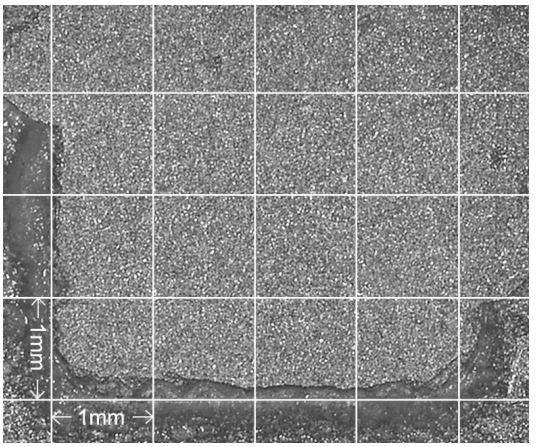

Figure 2A.

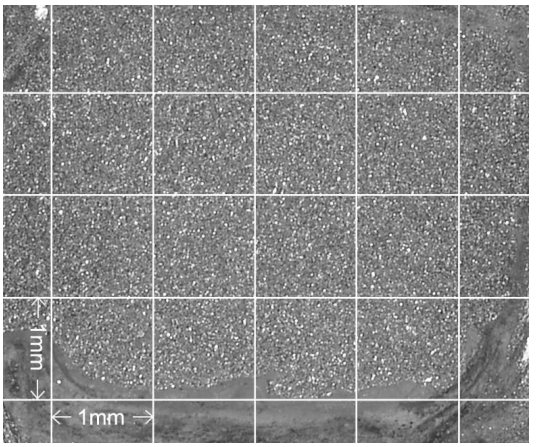

Figure 2B.

Figure 2. Representative samples of each group. **A**, AS group; **B**, AST group. An adhesive remnant index of 0 was most frequently observed in AS and AST groups.

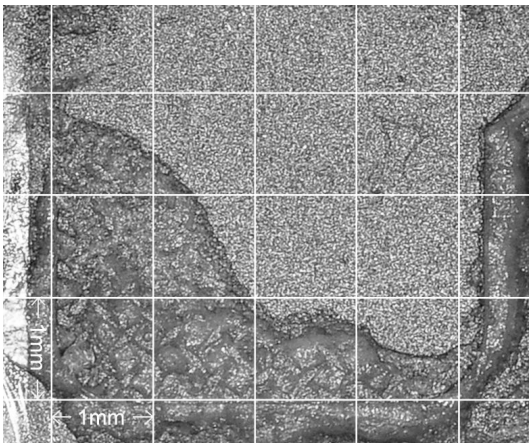

Figure 3A.

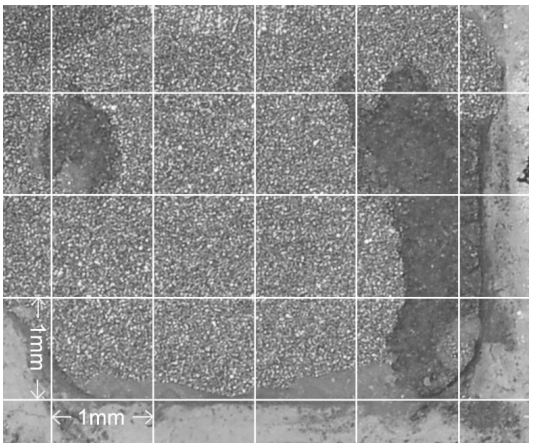

Figure 3B.

Figure 3. Representative samples of each group. **A**, MP group; **B**, MPT group. An adhesive remnant index of 1 was most frequently observed in MP and MPT groups.

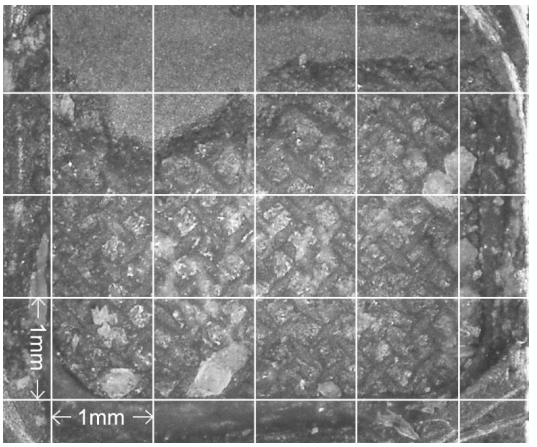

Figure 4A.

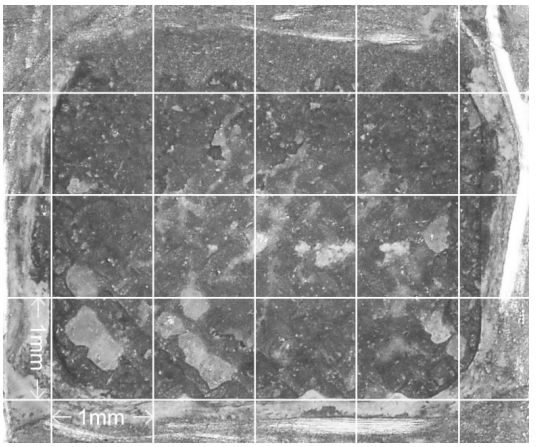

Figure 4B.

Figure 4. Representative samples of each group. **A**, SC group; **B**, SCT group. An adhesive remnant index of 2 was most frequently observed in SC and SCT groups.