

## Effects of sandblasting media and steam cleaning on bond strength of titanium-porcelain

Che-Shun WANG<sup>1</sup>, Ker-Kong CHEN<sup>2</sup>, Kiyoshi TAJIMA<sup>1</sup>, Yuki NAGAMATSU<sup>1</sup>, Hiroshi KAKIGAWA<sup>1</sup> and Yoshio KOZONO<sup>1</sup>

<sup>1</sup>Division of Biomaterials, Department of Oral Functional Reconstruction, Kyushu Dental College, 2-6-1 Manazuru, Kokurakita-ku, Kitakyushu 803-8580, Japan

<sup>2</sup>Department of Conservative Dentistry, Kaohsiung Medical University Hospital and College of Dental Medicine, Kaohsiung Medical University, 100 Shih-Chuan 1st Road, San-Ming District, Kaohsiung, Taiwan 80708, Republic of China

Corresponding author, Ker-Kong CHEN; E-mail: enamel@kmu.edu.tw

The effects of sandblasting media and steam cleaning treatment after sandblast were examined on tensile bond strength of porcelain to titanium. The use of the commercially available silica-coated alumina particles for sandblast was significantly effective for increasing bond strength than the conventional alumina. It might be due to the increased surface roughness and existence of remaining silica on titanium surface. Additional application of the steam cleaning on titanium surface after sandblasting could make the surface configuration clear in SEM by removing some sandblasted particles loosely embedded in titanium as well as the debris and oily contaminants. The resultant bond strength was significantly improved to reach almost the maximum strength of this porcelain-titanium system regardless of the kind of sandblasting media used, which was confirmed by the observation of the failure mode showing that most of the fracture surface was occupied by cohesive failure in porcelain.

**Keywords:** Bond strength of titanium-porcelain, Sandblasting media, Steam cleaning

### INTRODUCTION

The great concern on a pleasing smile from patients makes aesthetics become an important issue in the restorative dentistry. Tooth-colored restorations are the first choice for most patients. Metal-porcelain and all porcelain prosthesis are two major restorations to fulfill patient's needs. All porcelain prosthesis, even good at color matching<sup>1</sup>, suffers from the problem of marginal discrepancy<sup>2</sup>. In fact, metal-porcelains still enjoy wide acceptance and are commonly used restoration.

The fabrication of metal-porcelain restoration is a technical sensitive procedure, including: (1) fabrication of metal coping, (2) metal surface treatment for increasing bond strength, which is considered the most important step for the success of porcelains fused to metal in the whole procedure, (3) application and firing of porcelain onto the metal to complete the restoration.

Metal substrates have an effect on the physical properties of metal-porcelain restoration. The metals used in dental field could be classified into precious, semiprecious, and non-precious alloys. Non-precious alloys have the disadvantages of poor biocompatibility, low corrosion resistance, poor bond strength, easy discoloration of porcelain<sup>3,4</sup> and difficulty in fusing<sup>5</sup> that are not used so frequently. However, with the increasing requirement of dental implant in dental field, the need for metal-porcelains on top of implant is prevailing. Most of the implants and their abutments, no matter what kind of systems is used, are made of titanium and its alloy. Therefore, titanium-porcelain restorations play an increasing important role in the

modern dentistry, which could have the benefit of having two similar materials contacted directly. These might reduce the possibilities of corrosion and wear. Welander *et al.* also found the benefit that soft tissue healing is much better with titanium coping after implant loading than the other metal ones<sup>6</sup>.

Titanium and its alloys possess the characteristics of low specific gravity (4.52 g/cm<sup>3</sup>; while 18.3 g/cm<sup>3</sup> in high noble metal alloy for porcelain-fused-to-metal (PFM) restoration)<sup>7</sup>, biocompatibility<sup>8</sup>, high strength, high heat resistance, high corrosion resistance<sup>9</sup>, low cost, availability<sup>10</sup>, and additionally titanium has high dampening capacity, which could absorb impact force rapidly. These properties make titanium and its alloys capable of substitutes for noble metal alloys.

When titanium is used in PFM restoration, the bonding with porcelain needs more concern. The disadvantages of titanium are: (1) high melting point (1,668°C; while 1,063°C in gold<sup>7</sup>); (2) strong affinity to oxygen, nitrogen and carbon at high temperatures<sup>11</sup>, which requires casting circumstance to be in a vacuum or inert gas chamber; (4) poorly adherent oxide layer if heated above 883°C<sup>12,13</sup>, and (5) use of low fusing porcelains<sup>14</sup>. In 1959, King *et al.* concluded that intimate contact between the metal and porcelain can be achieved by increasing wettability of the metal surface<sup>15</sup>, which can be made by increasing surface energy. Sandblasting with alumina (Al<sub>2</sub>O<sub>3</sub>) is one of the methods that are recommended for creating surface irregularities and providing mechanical interlocking force for porcelain. Derand *et al.*<sup>16</sup> and Lautenschlager *et al.*<sup>17</sup> found significant amounts of alumina particles embedded in the titanium-porcelain interface, which is

attributed to the sandblasting surface treatment. The presence of such particulates may have an adverse effect on weakening the mechanical interlocking of porcelain to metal by introducing stress concentration points<sup>16,17</sup>.

Improving the bond strength of porcelain to titanium is important for enhancing its clinical usability. However, the bonding between titanium and porcelain is always an important issue. Yilmaz *et al.* investigated the bond compatibility between porcelain and titanium and found that bond compatibility between titanium and porcelain was only comparable with the NiCr-porcelain system<sup>18</sup>. Surface treatment with sandblasting or sandblasting combined with ultrasonic cleaning pretreatment to titanium is suggested to effectively enhance the bond strength with porcelain<sup>19,20</sup>. Steam treatment is one of the methods in laboratory procedure to remove residue and debris before porcelain application, which is recommended to be used as an alternative to ultrasonic cleaning in dental laboratory<sup>21</sup>. Jochen mentioned that the additional surface treatment of aluminum oxide and steam cleaning could increase the bond strengths of porcelain to gold-palladium alloy<sup>22</sup>, while Graham reported a reverse effect of the steam cleaning onto the bond strength<sup>23</sup>. In fact, there is still little information concerning the influence of the surface pretreatment of steam cleaning, sandblasting media or the combination of them on enhancing the bond strength of titanium and porcelain.

This study was, therefore, conducted to investigate the method of increasing the bond between titanium and porcelain by the use of different sandblast powders and steaming treatment on the titanium surface in

order to find out the optimal method for improving the bond strength of titanium and porcelain.

## MATERIALS AND METHODS

In this study, a commercially fabricated pure titanium (ST-50 CpTi, JIS Class 2, Sumitomo Metal, Tokyo, Japan) in diameter of 8 mm was used. Each titanium rod in 15 mm length was prepared with one end of the rod polished with SiC sandpaper in the sequences of grit-240, 500, and 1000 to serve as a specimen.

The titanium specimens were divided into eight groups as follows according to the surface treatment before porcelain powder application:

- (1) N-CONT group, as polished without sandblasting and steam cleaning (served as a control),
- (2) N-A050 group, sandblasted with 50  $\mu\text{m}$  alumina powder (HI Aluminas, Shofu, Kyoto, Japan) for 20 seconds at a pressure of 0.2 MPa and at a distance of 10 mm between the nozzle and the surface, while no steam cleaning was applied,
- (3) N-A125 group, sandblasted with 125  $\mu\text{m}$  alumina powder (HI Aluminas, Shofu, Kyoto, Japan) for 20 seconds at a pressure of 0.2 MPa and at a distance of 10 mm between the nozzle and the surface, while no steam cleaning was applied,
- (4) N-ROCT group, sandblasted with 110  $\mu\text{m}$  silica-coated alumina powder (Rocatec Plus<sup>®</sup>, 3M ESPE, USA) for 20 seconds at a pressure of 0.2 MPa and at a distance of 10 mm between the nozzle and the surface, while no steam cleaning was applied,
- (5) S-CONT group, application of steam cleaning (Steam Cleaner JS-2500, Sanyo, Osaka, Japan) for one minute was performed onto the specimens of N-CONT

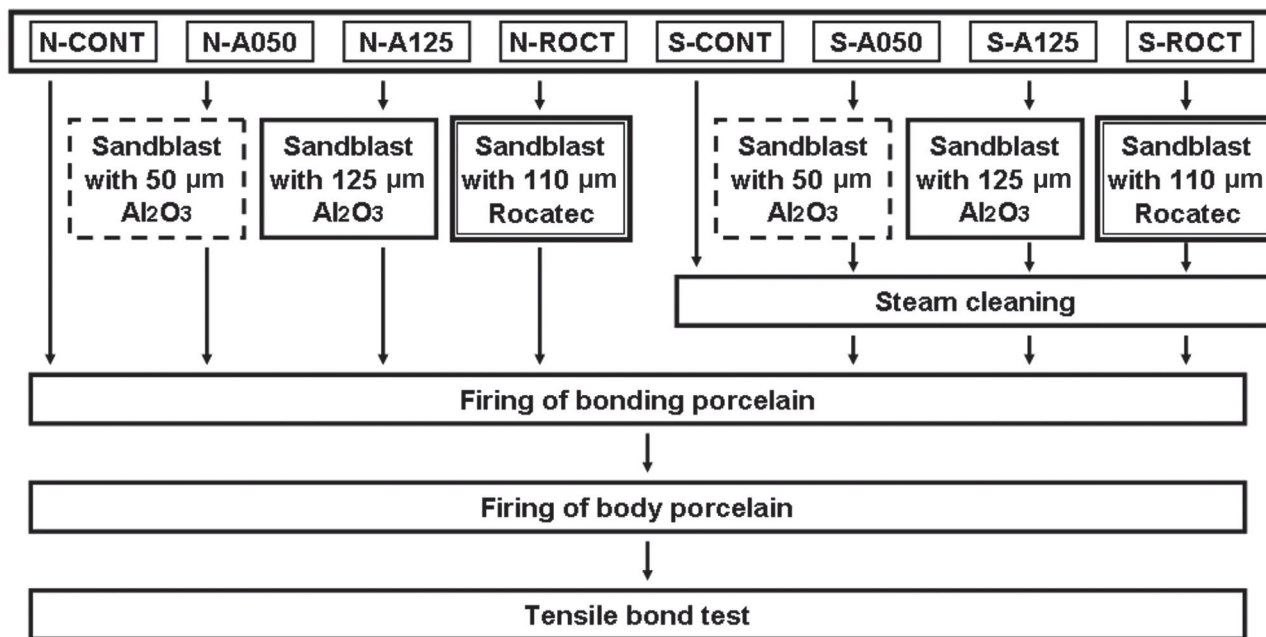


Fig. 1 Diagram of the whole detail procedures for preparing a tensile bond test specimen.

group,

(6) S-A050 group, application of steam cleaning for one minute was performed onto the specimens of N-A050 group,

(7) S-A125 group, application of steam cleaning for one minute was performed onto the specimens of N-A125 group,

(8) S-ROCT group, application of steam cleaning for one minute was performed onto the specimens of N-ROCT group.

Oxidization heat treatment was not performed to avoid unexpected incorporation of scattered bond strengths according to a report of Ohara<sup>24</sup>) that a thin oxide film existing on the as-polished titanium surface would be preferable rather than a thick brittle TiO<sub>2</sub> formed by heat treatment.

The procedure from different surface treatment of titanium, pile of bonding porcelain, pile of body porcelain till completion of a testing specimen was shown in Fig. 1.

Ten specimens were assigned for each group. The surface roughness, surface structure and element distribution on each surface-treated titanium specimen in each group were analyzed with a profilometer (Surfcorder SE-2300, Kosaka Laboratory Ltd., Tokyo, Japan), a scanning electron microscope (S-3000N, Hitachi, Tokyo, Japan) and an energy-dispersive X-ray microanalyzer (SE4300, Hitachi; EMAX-550, Horiba, Japan), respectively. Each specimen was measured three times on different areas.

After surface treatment, bonding porcelain (Super Porcelain Ti-22, Noritake, Nagoya, Japan) with 0.2 mm in thickness was applied on one end of titanium specimen and fired with a firing furnace (IVOCLAR Programat P100, Evoclar Vivadent, Tokyo, Japan)

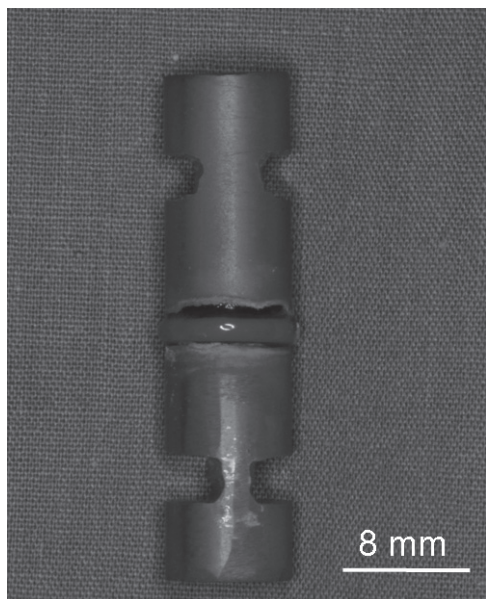


Fig. 2 A completed fired porcelain-titanium specimen for tensile bond test.

according to the manufacture's instruction. Body porcelain was then applied on the bonding porcelain between two titanium specimens in a way of butt joint and the thickness of the porcelain layer was adjusted to 1.5 mm in total before firing leaving a little amount of overflow to compensate for the firing shrinkage. The excessive porcelain after firing was ground with a fine diamond point paying careful attention not to form notches and the specimen was finished by glazing under atmosphere (Fig. 2).

A universal testing machine (Tensilon UTM-III-500, Shimadzu, Kyoto, Japan) was used to measure the tensile bond strength at a crosshead speed of 2.0 mm/min under the temperature of 25±2°C. The fracture surfaces of each specimen were examined and photographed by the light microscope at 30X magnification. The area over which each failure mode occurred in each specimen was measured by the use of NIH Image J software<sup>25</sup>) and the sum of the same failure mode was calculated to convert into percentage to reveal the occupational ratio of each failure mode in each group.

All of the data were statistically analyzed with one-way ANOVA, followed by Fisher's PLSD test using an Excel add-in software (Statcel)<sup>26</sup>) to find out the significance among those groups.

## RESULTS

### Surface roughness of titanium specimen

Regardless of steam cleaning, surface roughness (*Ra*) of titanium specimen showed the lowest value in CONT groups and increased in the sequence of A050 groups, A125 groups and ROCT groups (Fig. 3). The comparison of A050 groups and A125 groups revealed that larger alumina particle presented a significantly larger *Ra* value ( $p < 0.05$ ). All groups showed no

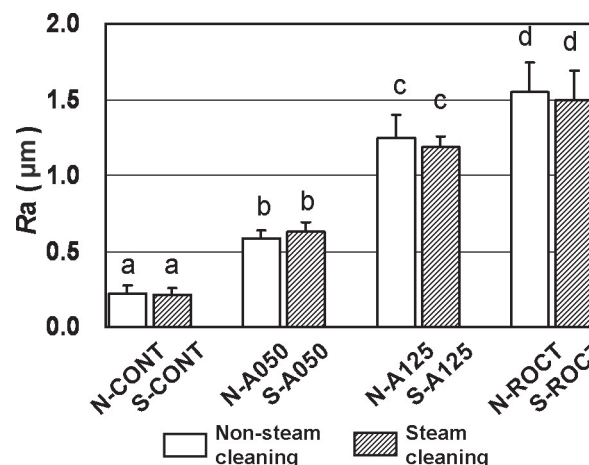


Fig. 3 Surface roughness of titanium specimen after different surface treatment. Different letters indicate statistically significant differences ( $p < 0.05$ ). The error bar shows SD ( $n = 10$ ).

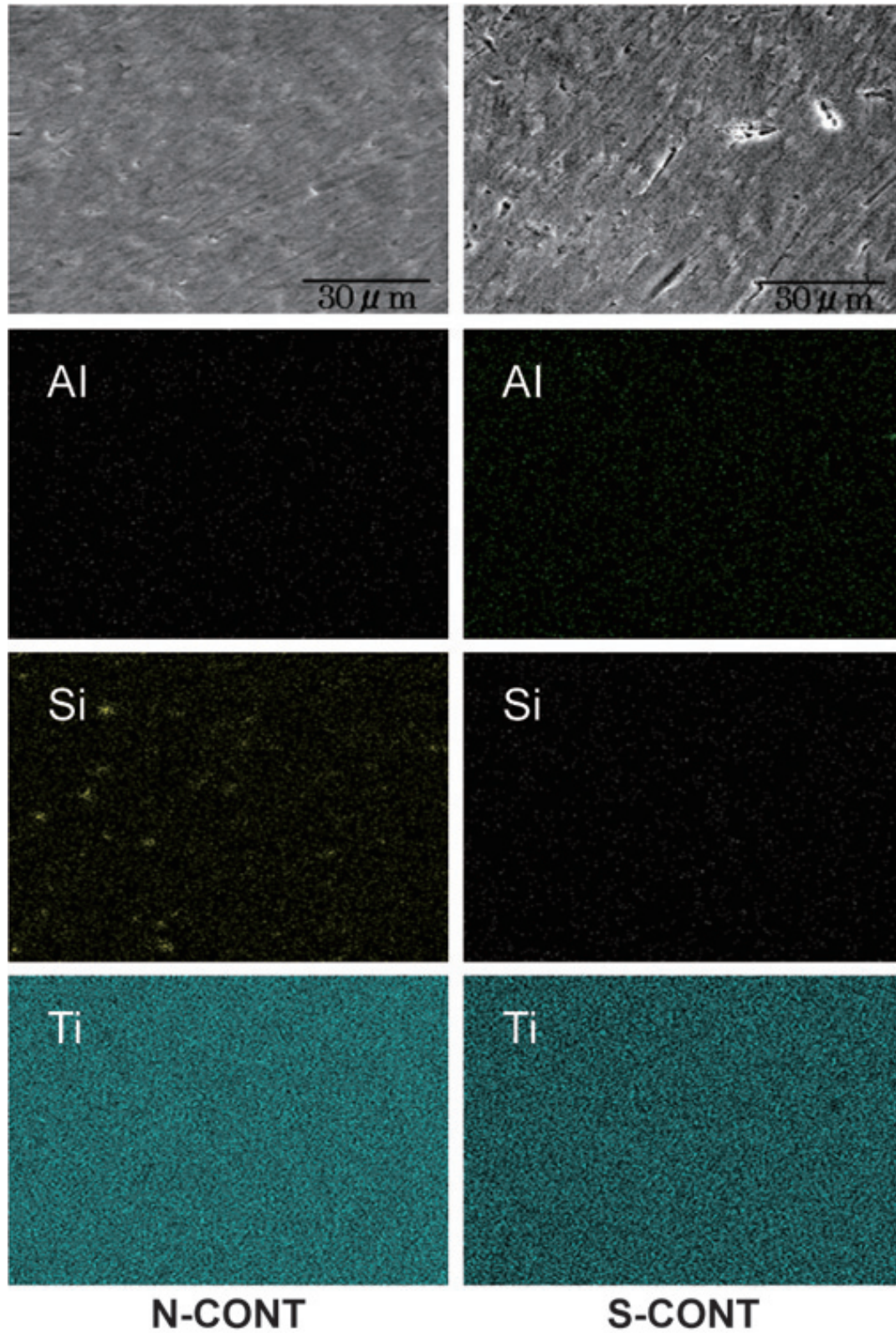


Fig. 4 SEM images (top) and X-ray images showing mapping of Al, Si, and Ti elements on titanium surface in N-CONT (left) and S-CONT (right) groups.

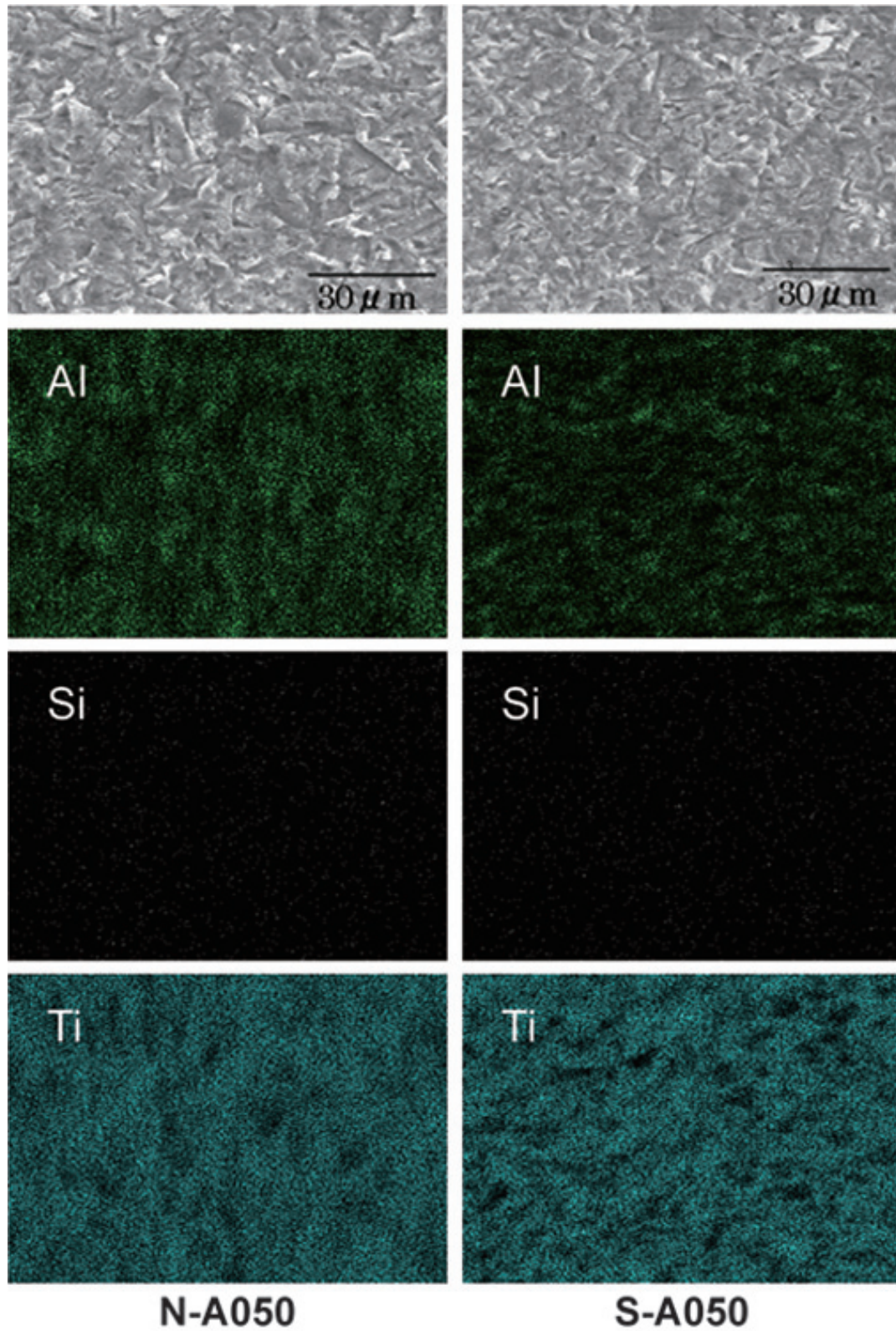


Fig. 5 SEM images (top) and X-ray images showing mapping of Al, Si, and Ti elements on titanium surface in N-A050 (left) and S-A050 (right) groups.

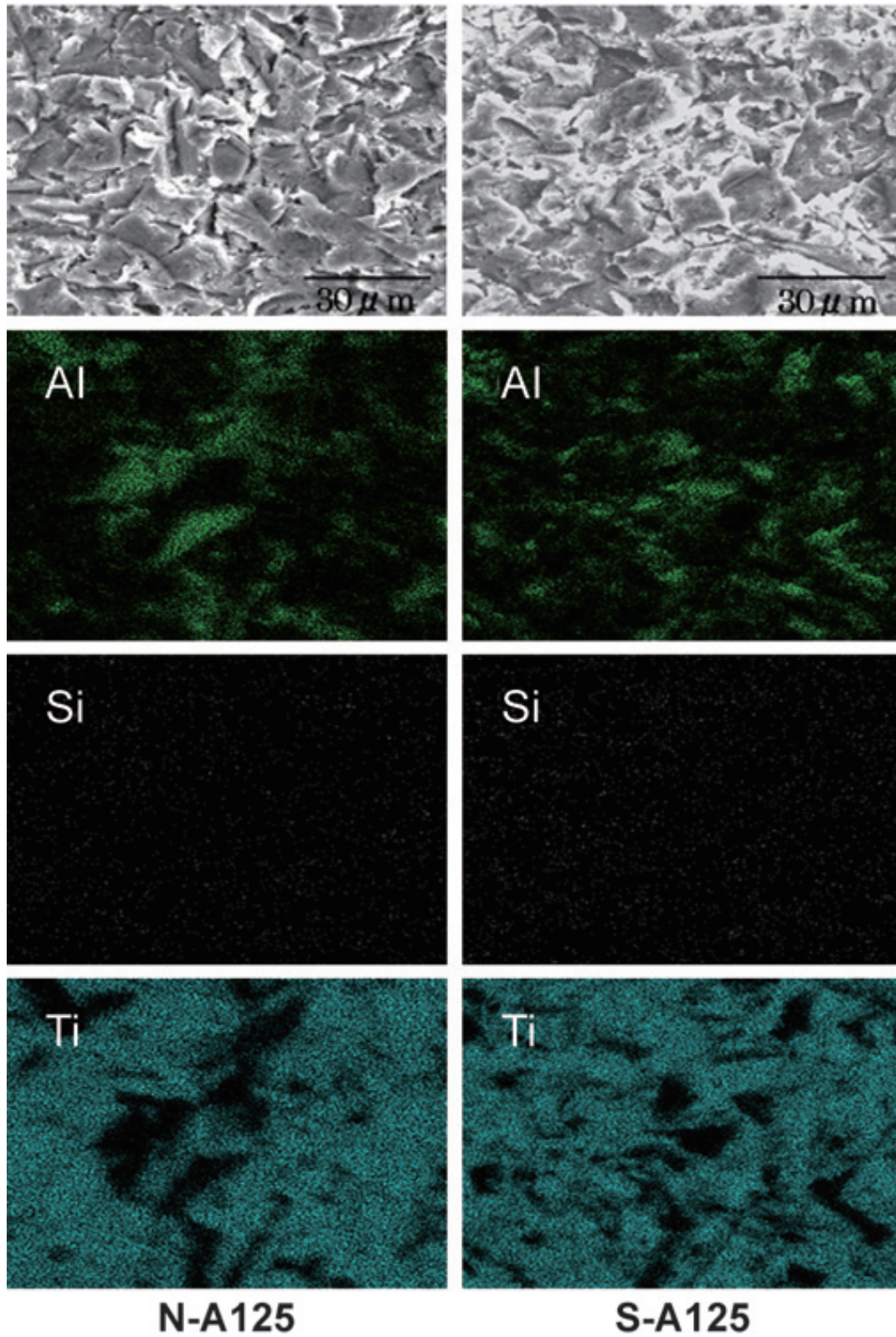


Fig. 6 SEM images (top) and X-ray images showing mapping of Al, Si, and Ti elements on titanium surface in N-A125 (left) and S-A125 (right) groups.

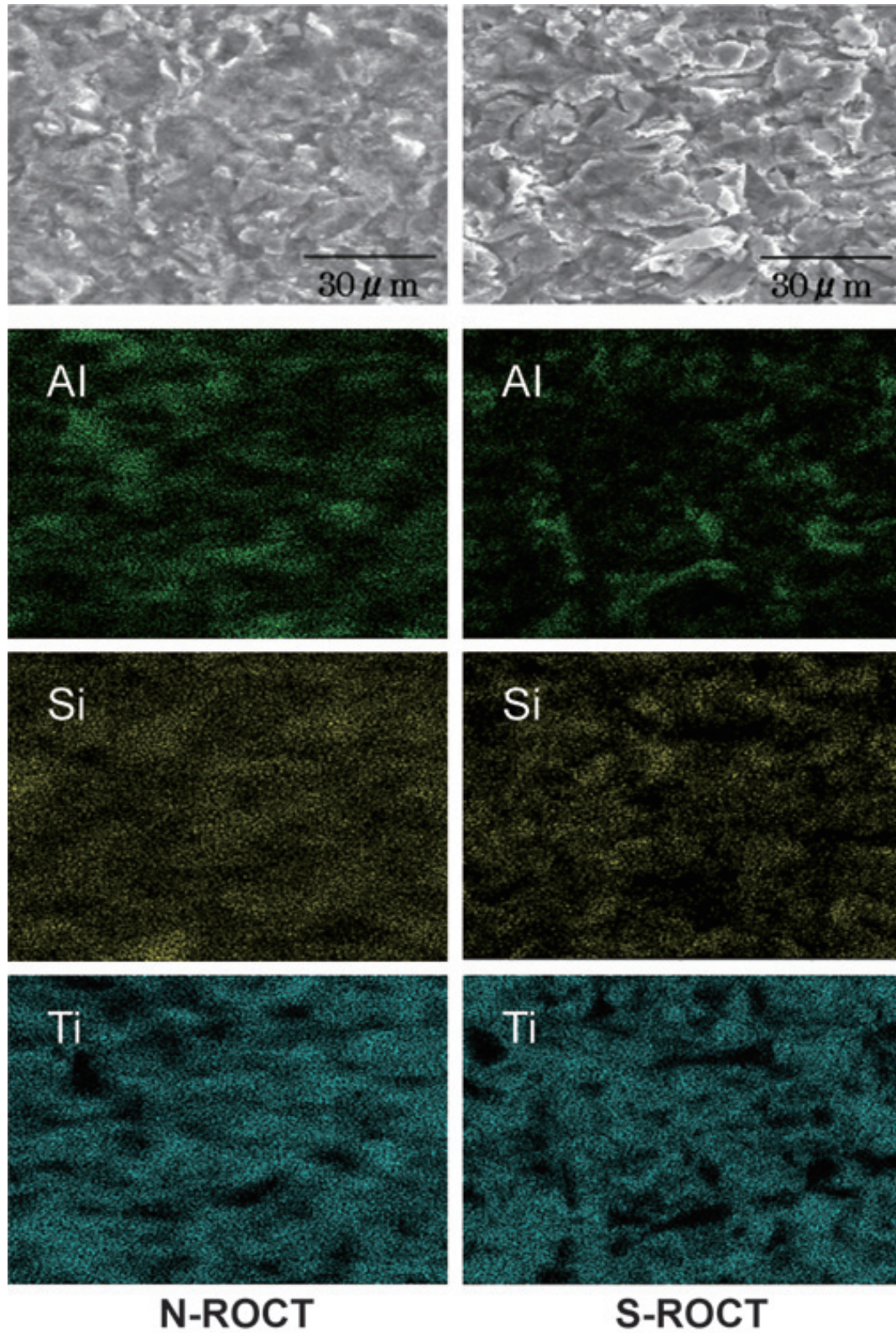


Fig. 7 SEM images (top) and X-ray images showing mapping of Al, Si, and Ti elements on titanium surface in N-ROCT (left) and S-ROCT (right) groups.

Table 1 X-ray microanalysis of titanium surface in each group (%)

Steam	Element	CONT	A050	A125	ROCT
N	Al	0.0 (0.0)	21.2 (1.3)	17.0 (4.2)	18.0 (1.5)
	Si	0.3 (0.1)	0.1 (0.1)	0.4 (0.1)	16.8 (1.3)
	Ti	99.7 (0.1)	78.8 (1.3)	82.6 (4.2)	65.2 (2.6)
S	Al	0.0 (0.0)	22.3 (0.8)	16.9 (0.9)	16.5 (2.7)
	Si	0.2 (0.1)	0.0 (0.1)	0.1 (0.1)	8.5 (1.5)
	Ti	99.8 (0.1)	77.7 (0.8)	83.2 (0.9)	74.9 (4.0)

$n=10$ /group; mean (SD)

N: non-steam cleaning, S: steam cleaning

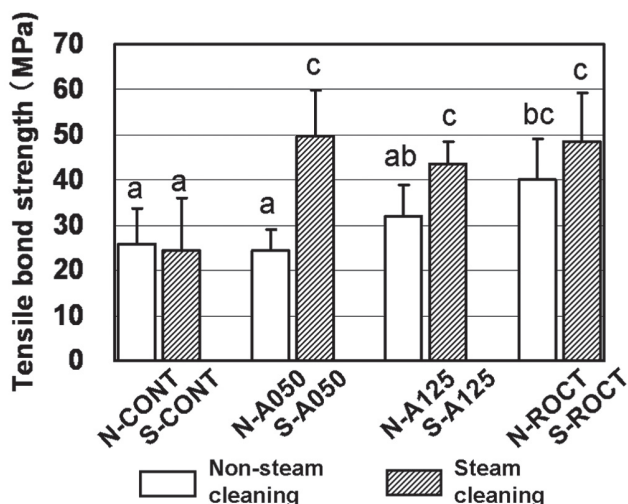


Fig. 8 Tensile bond strength of each group. Different letters indicate statistically significant differences ( $p<0.05$ ). The error bar shows SD ( $n=10$ ).

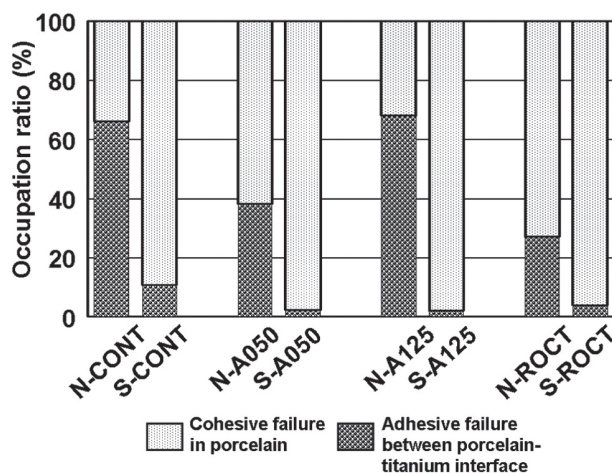


Fig. 9 The failure mode in each group.

significantly difference in surface roughness between with and without steam cleaning treatment.

#### Observation of sandblasted titanium surface

The SEM pictures of the treated surface in each group were shown in the upper row of Figs. 4 to 7. Sandblasted groups, regardless of the media or steam cleaning, showed prominent irregular surface as compared with control group. It showed more apparent irregularity in A125 group than A050 group. Among the different sandblasting media, ROCT group showed similar surface characteristics as A125 group. The steam cleaning groups presented somewhat clear image as compared with the non-steam cleaning groups.

#### X-ray microanalysis of titanium surface

Al, Si, and Ti were the target elements focused on analysis. The percentage of the existence of each element and its mapping in each group were shown in Table 1 and Figs. 4-7, respectively.

#### Al content:

Al showed no existence in CONT group, while

increased after sandblast treatment irrespective of the media used. The order of Al contain was in the order of  $A050 > A125 \geq ROCT > CONT$ . Steam cleaning showed no significant effect on Al variation.

#### Si content:

Si showed the highest value in ROCT group, while few in other groups. Each steam cleaned group showed a decline tendency of Si, especially prominent in ROCT group.

#### Ti content:

Ti was highest in CONT group, while lowered down by the increase of Al and Si in other groups. The steam cleaning did not show significant change to each group.

#### Tensile bond strength

The tensile bond strength of eight groups was shown in Fig. 8. In the bond strength of the non-steam cleaning, there was a significant difference between N-CONT and N-ROCT ( $p<0.05$ ) while no significant differences could be found among other groups. The treatment of steam cleaning increased the tensile bond strength in



all the sandblasted groups: S-CONT, S-A050, S-A125 and S-ROCT, showing significant differences between S-CONT and the sandblasted groups ( $p < 0.05$ ).

According to the result of Fisher's PLSD, the significant differences could be found in total between the non-steam cleaning groups and the steam cleaning groups ( $p < 0.05$ ), and between the control group and the sandblasted groups ( $p < 0.05$ ).

#### Failure mode observation

Almost all the specimen showed mixed failure mode involving cohesive failure in porcelain and adhesive failure at the interface of titanium and porcelain in tensile test. The average occupations of the cohesive failure in porcelain and adhesive failure appeared in the projected area of the fracture surface were calculated and shown in Fig. 9. The ratio of the cohesive failure in porcelain was significantly increased by steam cleaning in any group. Especially the most part of the fracture surface was occupied by the cohesive failure in porcelain in the steam-cleaned groups regardless of the sandblasting media.

## DISCUSSION

The sandblasting technique with alumina particles has been commonly employed for many purposes in dentistry, including (1) removal of contaminants (2) increase of effective surface area<sup>27</sup> and (3) improvement of wetting ability of porcelain<sup>28</sup>. Lavine *et al.* found that cast surface roughened by stone could increase the bond strength of porcelain to metal as compared with non-roughened one due to the resultant increased surface area, which improved the wettability of metal by diffusing the porcelain particles into the metal-porcelain interface<sup>29</sup>. It was also reported that the use of a large particle size of alumina was advantageous in increasing the surface roughness and promoting mechanical interlocking of titanium with porcelain<sup>30,31</sup>. When the titanium surface was sandblasted with alumina particles in the present study, the surface roughness was significantly increased (Fig. 3) and the bond strength of porcelain to titanium tended to increase (Fig. 8). However, no statistically significant differences were detected in bond strength among N-CONT, N-A050 and N-A125, although the surface roughness increased as the particle size of alumina increased. Another factor affecting the bond strength seemed to be the amount and behavior of alumina particles embedded in titanium. The X-ray microanalysis revealed the existence of alumina particles after sandblasting (Table 1). It is known that the alumina particles are embedded into titanium by the process of sandblasting<sup>16,17,24</sup>. The existence of these particles may sometimes have a favorable effect<sup>24</sup> or an adverse effect<sup>16,17</sup> on the bond strength probably depending on their fixation in the matrix metal. If the particles are loosely embedded in titanium, the porcelain will be peeled off from titanium surface by accompanying the particles. It may lead to the

increased adhesive failure at the interface between porcelain and metal. The tightly fixed particles, on the contrary, will give an effect of interlocking and enhance the bonding, resulting in decreased adhesive failure. From the fact that the occupation ratio of adhesive failure was significantly larger in N-A125 than in N-A050, it is estimated that there were not a few loosely fixed alumina particles on titanium surface before steam cleaning. This might be the reason why N-A125 showed not significantly larger bond strength than N-A050 in spite that significantly larger surface roughness was created in the former group.

ROCT group was sandblasted with aluminum oxide particle coated with a thin layer of silica (Rocatec Plus<sup>®</sup>). It was reported that the microblast with these particles could form a tribochemical coating on the surface of the adherend surface, which might be of resin, porcelain or metal, resulting in the improvement of the bonding with resin<sup>32,34-38</sup>. It has the advantage of forming "cold silicatisation" to prevent the thermal stressing within the metal and avoid distortion. N-ROCT group showed a prominently irregular surface structure in SEM observation (Fig. 7) with significantly larger surface roughness than alumina sandblasted groups. Due to these effects of increased surface area and interlocking, N-ROCT group showed the largest bond strength among the non-steam cleaning groups. The outstanding Si content was found on the titanium surface by X-ray microanalysis (Table 1) indicating the existence of the embedded particles or trace of silica on the concave wall after the particle had removed as the manufacturer of the particle claims. Anyway, the presence of silica might give an additional enhancing effect on the bonding of porcelain to titanium. This result confirmed that Rocatec Plus<sup>®</sup> is an available sandblasting media to improve the bond strength of porcelain to titanium as shown in the earlier reports for composite<sup>33-35</sup>.

It was reported that ultrasonic cleaning of sandblasted and tribochemically silica-coated titanium would be to improve resin bonding as loose surface particles could be removed without relevant change in composition, the element of alumina or silica<sup>33</sup>. Steam cleaning after sandblast pretreatment of titanium is simple and easy method to remove the debris, oil etc. on the metal surface before piling porcelain. It was found in Figs. 4 to 7 that the surface configurations as well as the distribution of each element were made clear by steam cleaning. This may indicate that the debris and some loosely attaching particles used for sandblast were removed by the cleaning treatment. However, it could not be confirmed by X-ray microanalysis, in which no significant changes in Al content were detected after steam cleaning treatment regardless of the kinds of sandblast media. Although X-ray microanalysis is originally available for a quantitative analysis on smooth surface, it cannot but be regarded as a semi-quantitative analysis in this experiment since it was performed on irregular surface. Therefore, it seemed meaningless to compare the small

change in quantity in Table 1. Besides, the elements can be detected from the surface layer about 10 $\mu$ m in depth and some alumina particles might have been ignored if they lay under other particles in non-steam cleaning group. Exceptionally a marked reduction in Si to half of N-ROCT group was found by steam cleaning. This was obviously resulting from the removal of silica and it appears that the silica has been impregnated into the titanium not so firmly as the report of Kern<sup>33</sup>.

The steam cleaned groups after sandblasted with alumina, S-A050 and S-A125, showed significant increase in bond strength compared with as-polished or as-sandblasted groups while there was no statistical difference between S-A050 and S-A125. The steam cleaning caused no changes in surface roughness but a characteristic features in the failure mode by tensile testing. Fracture occurs at the weakest structure in the system. Almost all the specimens failed in the mixed failure mode involving cohesive failure in porcelain and adhesive failure at the interface of porcelain and titanium and their occupation ratios were characterized by the steam cleaning treatment. Although they were competed with each other before steam cleaning, adhesive failure markedly reduced and majority of the fracture surface was occupied by cohesive failure in porcelain after the specimen was subjected to steam cleaning (Fig. 9). Such a reduction of the adhesive failure means that the bonding ability of porcelain to titanium might exceed the strength of porcelain itself, and the bond strength of this system might reach almost the maximum level in tensile testing.

In N-ROCT group, the largest bond strength was obtained among the non-steam cleaned groups. Since it was already at the maximum level as large as S-A050 and S-A125, no more significant increase in bond strength could be expected after steam cleaning treatment although adhesive failure significantly decreased. Hence, the experimental eight groups could be clearly classified into two categories in failure mode depended on the application of steam cleaning: (1) non-steam cleaning group: high ratio of adhesive failure at the interface of porcelain and titanium, and low ratio of cohesive failure in porcelain; (2) steam cleaning group: extremely low ratio of adhesive failure, and high ratio of cohesive failure in porcelain. It indicates that such a characteristic failure mode was closely related with bond strength after sandblasting and the additional application of steam cleaning had a positive influence on bond strength between porcelain and titanium with less adhesive failure.

The steam cleaning also decreased the occupation ratio of adhesive failure in CONT group. Little amount of Si was detected in control group even though the pure titanium specimen received no sandblasting pretreatment. The preparation of a titanium specimen was performed by grinding under water with sandpaper, which contains particles of SiC. There is a high possibility that SiC particle might be incorporated

into the titanium surface and remained left during the preparation procedure. It is consistent with the result of Miyakawa<sup>23</sup>. The decreased adhesive failure may indicate that the loosely fixed SiC particles were removed by steam cleaning. However, the adhesive failure still occupied about 10% of the fracture surface and the cleaning effect could not be reflected on the improvement in bond strength because of the potential initiation of crack or absolute lack of interlocking at the porcelain-titanium interface.

From the above findings, it is strongly suggested that the bond strength of porcelain-titanium can be extremely improved by the application of sandblasting with silica-coated alumina (Rocatec Plus<sup>®</sup>) as well as the additional treatment of steam cleaning following sandblast regardless of the sand media. It can be, therefore, recommended in practical use of titanium for PFM restoration with maximized bond strength to sandblast with silica-coated alumina and additionally apply the steam cleaning. Especially when the conventional alumina particles are used for sandblasting, the addition of the steam cleaning may be advisable for increasing the bond strength.

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