Effect of immediate dentin sealing applications on bonding of CAD/CAM ceramic onlay restoration

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The effects of immediate dentin sealing (IDS) applications on the bonding of computer-aided design/computer-aided manufacturing (CAD/CAM) ceramic onlay restorations after cyclic loading were examined. Standardized mesial-distal-occlusal-palatal cavities in 32 extracted human molars were prepared. The cavities were divided into four groups: those receiving thin-layered (T), slope-shaped (S), and base-shaped (B) sealing, and the non-sealing group (N) as a control. The intra-cavity dentin walls of the T, S, and B groups were sealed with an all-in-one adhesive and a flowable composite. All cavities were scanned; hence, CAD/CAM onlays were fabricated using ceramic blocks and bonded with a resin cement system. Cyclic loading was applied and the microtensile bond strength (μ-TBS) was measured. It was found that IDS application improved not only the μ-TBS, but also the bonding reliability and durability of the CAD/CAM restoration. In particular, the S restoration exhibited the highest-performance as regards both robust bond strength and stable bonding.

Keywords: Immediate dentin sealing application, CAD/CAM ceramic restoration, Cyclic loading, Microtensile bond strength, Weibull analysis

INTRODUCTION

Esthetic restoration is one of the patient’s earnest desires. Ceramic restoration offers to the patients to fulfill their desires. To date, excellent clinical success has been achieved for bonded ceramic inlay and onlay restorations3), and the associated technique has been evaluated as having an acceptable operative modality20). Machinable ceramic restoratives as used by the CEREC system yield a satisfactory restoration with an acceptable marginal adaptation and clinical longevity4), along with a high success rate, color stability, and wear resistance41). As regards disadvantages, indirect restoration may cause post-operative sensitivity and result in bacterial invasion, because of dentinal tubule exposure due to cavity preparation3). Thus, the application of immediate dentin sealing (IDS) using flowable resin composite is recommended to protect the exposed dentin surfaces of the cavity walls after preparation6,7). The applied IDS then generates a dentin hybrid layer and provides an integrated sealing coat on the dentin surface. In vitro studies have indicated that IDS improves the bonding of resin cement significantly6,8,9) and decreases the thermal diffusion of the restoration10). In addition, IDS has been found to improve not only the marginal leakage prevention11), but also the cavity adaptation of indirect restorations12,13). Further, a randomized controlled in vivo clinical trial study14) over a 24-month period showed a significant decrease in the occurrence of postcementation hypersensitivity for the IDS group compared to the non-IDS group.

However, the effect of clinical IDS application involving a variety of IDS thicknesses and shapes on the bonding of computer-aided design/computer-aided manufacturing (CAD/CAM) ceramic restoration has not been examined. Bond strength measurement is a typical indicator of the status of an adhesive restoration. In particular, the microtensile bond strength (μ-TBS) test15) is an effective means of measuring intra-cavity bond strength before and after simulated clinical stress. In addition, Weibull analysis is an appropriate statistical method to characterize the bonding outcome16). The purpose of the present study was to examine the effect of different IDS applications on the bonding of CAD/CAM ceramic onlay restoration after cyclic loading simulating an intra-oral environment, through measurement of the μ-TBS, investigation of the bonding reliability and durability, and observation of the failure mode. The null hypotheses of this study were as follows: 1) The IDS application does not affect the resin cement layer thickness; 2) The IDS application does not influence the μ-TBS, bonding reliability, and durability.

MATERIALS AND METHODS

Experimental materials

Table 1 lists the composition, lot number, and manufacturer of each material used in this study. For IDS materials, a typical all-in-one adhesive system (Scotchbond Universal Adhesive, 3M ESPE, Seefeld, Germany), and a popular low-viscosity restorative composite (Filtek Supreme Ultra Flowable Restorative, 3M ESPE) were used. A well-known chair-side CAD/CAM system comprised of a CEREC AC Omnicam (SW v4.3, Dentsply Sirona, Bensheim, Germany) and CEREC MC XL (Dentsply Sirona) was used to design and fabricate the restoratives. For the dental CAD/CAM restorative block, the most popular feldspathic ceramic block, i.e.,
### Table 1 Materials used

| Immediate dentin sealing materials | Composition                                                                                      | Lot no. | Manufacturer |
|-----------------------------------|-------------------------------------------------------------------------------------------------|---------|--------------|
| Scotchbond Universal Adhesive     | MDP, Dimethylacrylate resins, HEMA, Methacrylate-modified polyalkenoic acid copolymer, Filler, Ethanol, Initiators, Silane, Water | 569479  | 3M ESPE      |
| Filtek Supreme Ultra Flowable Restorative (Shade: A3B) | Bis-GMA, UDMA, TEGDMA, 0.1–5.0 μm ytterbium trifluoride filler, Non-agglomerated/non-aggregated 20/75 nm silica filler, Aggregated 0.6–10.0 μm zirconia/silica cluster filler | N722873 |               |

### CAD/CAM system

- **CEREC AC Omnicam**
  - CEREC operating system software, version 4.3
  - Dentsply Sirona

- **CEREC MC XL**
  - CEREC operating system software, version 4.3
  - Dentsply Sirona

### Dental CAD/CAM restorative block material

- **VITABLOCS MarkII** (Shade: A3C)
  - Silicon dioxide, Aluminum oxide, Sodium oxide, Potassium oxide, Calcium oxide, Titanium dioxide
  - 44910 VITA

### Pretreatment materials for cementation

- **K-etchant gel**
  - 40% phosphoric acid, Colloidal silica, Water, Dyes
  - 2L0041 Kuraray Noritake Dental

- **Clearfil Ceramic Primer Plus**
  - 3-trimethoxysilylpropyl methacrylate, MDP, Ethanol
  - 650004 Kuraray Noritake Dental

- **PANAVIA V5 Tooth Primer**
  - MDP, HEMA, Hydrophobic aliphatic dimethacrylate, Accelerators, Water
  - 6Q0004 Kuraray Noritake Dental

### Adhesive resin cement

- **PANAVIA V5** (Universal Shade)
  - Paste A: Bis-GMA, TEGDMA, Silanated barium glass filler, Silanated fluoroalminosilicate glass filler, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Colloidal silica, Initiators, Accelerators
  - Paste B: Bis-GMA, Silanated barium glass filler, Silanated aluminium oxide filler, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Accelerators, Dl-Camphorquinone, Pigments
  - 7M0008 Kuraray Noritake Dental

MDP, 10-methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-GMA, bisphenol-A-glycidyl methacrylate.

VITABLOCS Mark II (VITA Zahnfabrik, Bad Säckingen, Germany), was selected. As regards the pretreatment materials prior to resin cementation, a phosphoric acid gel (K-etchant gel, Kuraray Noritake Dental, Tokyo, Japan) and a silane-coupling agent (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental) were applied to the inner surfaces of the fabricated onlays. Then, PANAVIA V5 Tooth Primer (Kuraray Noritake Dental) was applied to the intra-cavity surface in accordance with the manufacturer instructions. The PANAVIA V5 system-specific dual-cure adhesive resin cement (Kuraray Noritake Dental) was used for cementation.

For all-light irradiation in this study, a quartz-tungsten-halogen light unit (Optilux501, Kerr, Orange, CA, USA) in combination with a 13/8 mm turbo-chip, with an output of 700 mW/cm², was used. Before and after each light irradiation, the light intensity was measured and inspected using a radiometer (Demetron L.E.D. Radiometer, Kerr).

**Tooth selection and restorative procedures**

This study was approved by the Ethics Committee of the Nippon Dental University School of Life Dentistry, Tokyo (approval number: NDU-T2016-06). A total of 32 human caries-free extracted maxillary first molars of similar color and size were used, which were stored in 0.1% thymol solution at 24°C. The teeth were subjected to cavity preparation within one year after extraction.

Figure 1 shows a schematic flow chart of the experimental procedure. Each extracted human maxillary first molar was embedded in a standardized resin mold by establishing a plane settled with the three apexes of the buccomesial, buccodistal, and mesiopalatal cusps parallel to the base plane of the mold. An acrylic...
resin (Adfa, Shofu, Kyoto, Japan) was used for the embedding (Fig. 1-(a)). The non-prepared original crown form of each embedded molar was then scanned using a CEREC AC Omnicam and the scanned data were utilized to reproduce the crown form of each fabricated restorative (Fig. 1-(b)). Standardized mesial-distal-occlusal-palatal (MODP) cavities were prepared using a straight cylinder round-end diamond bur (FG107RD, ISO #: 198 090 023, mean grit size: 100 μm, Shofu) mounted in a custom-made cavity duplicator (Tokyo Giken, Tokyo, Japan). First, the pulpal wall was prepared at a depth of 3.0 mm from the deepest part of the central fossa; then, the mesiopalatal and distopalatal cusps were removed. The buccal wall was set at a distance of 2.0 mm from the central fossa; hence, standardized L-shaped MODP cavities were obtained (Fig. 1-(c)).

The standardized cavity specimens were then divided into four groups, labeled T, S, B, and N, each comprised of eight cavity specimens. For the T group, a thin-layered low-viscosity restorative sealing was applied from the dentino-enamel junction on the buccal wall to that on the pulpal wall, whereas for the S and B groups, a slope- and a base-shaped sealing were applied, respectively. The N group was comprised of non-sealed specimens and acted as a control (Fig. 1-(d)). First, Scotchbond Universal Adhesive was applied to the intra-cavity exposed dentin surfaces of the T, S, and B specimens, and these surfaces were then scrubbed for 20 s. After treatment, a gentle stream of air was blown over the liquid for approximately 5 s, until movement ceased and the solvent evaporated completely; then, the specimens were light-cured for 10 s. Thereafter, for the T specimens, 30 mg of a low-viscosity restorative (Filtek Supreme Ultra Flowable Restorative) was applied to the pretreated dentin cavity wall and the specimens were light-cured for 20 s. For the S specimens, 90 mg of flowable composite was applied to the pretreated dentin wall and the specimens were light-cured for 20 s. For the B specimens, the pretreated dentin wall was sealed in the same manner as for the S specimens; then, another 90 mg of flowable composite was applied to the surface of the slope-shaped IDS and the specimens were light-cured for 20 s. Prior to the experiment, mean requirement weight of flowable resin composite in mg, T; 30 mg, S; 90 mg and B; 180 mg, was obtained from the preliminary test for the three IDS type-restorations five each. The right weight of flowable resin composite was applied to the standardized cavity and shaped with a small blush. For the T, S, and B specimens, the intra-cavity enamel and dentin walls were then sectioned. Four standardized beam-shaped specimens were obtained from each restored specimen. The thicknesses of the IDS layer and resin cement layer were measured, and the μ-TBS was measured at a crosshead speed of 1.0 mm/min.
All cavity specimens were scanned using the CEREC AC Omnicam in accordance with the manufacturer instructions, regardless of restoration type. Scanned specimens were stored in 37°C water for approximately 30 min until the cementation of onlay to the specific cavity. Each MODP onlay was designed and fabricated using the scanned data of the original crown form, which was obtained before cavity preparation, using the bi-generic copy function of the CEREC MC XL (Fig. 1-(e)). The inner surface of the fabricated onlay was chemically cleaned with K-etchant gel for 5 s, before being rinsed and air-dried. Prior to cementation, Clearfil Ceramic Primer Plus was applied to the chemically cleaned inner surface of the onlay, which was then air dried through gentle air blowing. PANAVIA V5 Tooth Primer was applied to the intra-cavity surfaces of all specimens, regardless of restoration type, and allowed to react for 20 s, before air drying. Soon after, PANAVIA V5 resin cement was applied to the inner surface of each onlay, and the restorative was inserted into the relevant cavity under a constant seating force of 900 g for 1 min. The cement was light-cured for a total of 5 s using Optilux 501, by five directions, mesial, buccal, distal, palatal and occlusal, for 1 s each. The slightly hardened excess cement was carefully removed. Subsequently, the onlay was left for 3 min, with a constant seating force of 900 g being applied for 1 min (Fig. 1-(e)). The restored specimens were finished with a flame-type diamond point (DP-05, mean grit size: 60 μm; Kuraray Noritake Dental) and polished with a flame-type silicone point (1F, mean grit size: 45 μm; J Morita, Tokyo, Japan) in accordance with the manufacturer instructions. Then, the specimens were stored in 37°C water for 1 h (Fig. 1-(g)).

Cyclic load and μ-TBS testing
For each restored specimen, an opposing object was used for cyclic loading against the inner and outer inclined surfaces of the functional cusps of the fabricated CAD/CAM onlay. To prepare opposing object, a negative impression was made using an acrylic resin (Adfa, Shofu), with a custom-made reproduction device establishing the 3D positioning of the apparatus for cyclic loading. The negative-impression resin object was trimmed into an opposing object conforming to the inner and outer inclined surfaces of the partially remaining occlusal functional cusps. All specimens were subjected to cyclic loading stress with the opposing object in place, which was conducted at 157 N for 90 cycles/min and for 3×10⁵ cycles in total. This process was performed in 37°C water and controlled using a custom-made multifunction apparatus (Tokyo Giken, Fig. 1-(h)). After the cyclic loading, each restored specimen was sectioned using a water-cooled microtome (Leitz 1600 Saw Microtome, Ernst Leitz, Wetzlar, Germany). First, each restored specimen was sectioned buccopalatally three times, perpendicular to the cavity floor and buccal wall simultaneously, at 1.0 mm intervals. The obtained sections were labeled ①, ②, and ③, as shown in Fig. 1-(i). The specimen was then cut mesiodistally three times, simultaneously perpendicular to the cavity floor and parallel to the buccal wall; the resultant sections were labeled ④, ⑤, and ⑥, as shown in Fig. 1-(j). Four standardized beam-shaped specimens with cross-sectional areas of 1.0×1.0 mm² were obtained from each restored specimen: (1) mesiobuccal, (2) distobuccal, (3) mesiopalatal, and (4) distopalatal region specimens (Fig. 1- (k)). The thicknesses of the IDS layer and resin cement layer at the central parts of sectioned surfaces ① and ④ were measured using a light microscope (Measurescope MM-1200, Nikon, Tokyo, Japan, Fig. 1-(l)). Further, the μ-TBS of each test specimen was measured at a crosshead speed of 1.0 mm/min using a universal testing machine (Autograph AG-1, Shimadzu, Kyoto, Japan, Fig. 1-(m)). If any test specimens failed during trimming before the μ-TBS test (pre-testing failure; ptf), those specimens were recorded as having μ-TBS values of 0 MPa and included in the statistical analysis.

Statistical analysis
The differences in IDS layer thickness (n=16) between the buccal ((1) and (2) shown in Fig. 1-(k)) and palatal region specimens ((3) and (4), Fig. 1-(k)), and also between the mesial ((1) and (3), Fig. 1-(k)) and distal region specimens ((2) and (4), Fig. 1-(k)), were analyzed for each IDS restoration type using a paired Student’s t-test. The level of significance was set to 0.05 and Excel 2010 software for Windows (Microsoft, Redmond, WA, USA) was employed.

To investigate the differences in IDS layer thickness (n=16) among the three IDS restoration types (excluding the N restoration) and for each regional specimen (buccal: (1) and (2); palatal: (3) and (4); mesial: (1) and (3); and distal region specimens: (2) and (4); Fig. 1-(k)), the Kruskal-Wallis test was performed, followed by multiple comparison testing using the Steel-Dwass test with the level of significance set to 0.05. The Bonferroni adjustment was employed, along with IBM SPSS statistics software (v19.0, IBM, Armonk, New York, USA).

The differences in resin cement layer thickness (n=16) between the buccal ((1) and (2)) and palatal region specimens ((3) and (4)), and also between the mesial ((1) and (3)) and distal region specimens ((2) and (4)), were analyzed for each IDS type using a paired Student’s t-test. Again, the level of significance was set to 0.05 and Excel 2010 for Windows (Microsoft) was used. To investigate the differences in resin cement layer thickness (n=16) among the four restoration types and within each regional specimen, a one-way analysis of variance (ANOVA) test and Tukey’s honest significant difference (HSD) test were performed, with the level of significance again set to 0.05 and using Excel 2010 for Windows.

In addition, differences in μ-TBS (n=16) between the buccal and palatal region specimens and also between the mesial and distal region specimens, were analyzed for each restoration type using a paired Student’s t-test, with the same level of significance and software as previously. To examine the differences
in μ-TBS \((n=16)\) among the four restoration types, for each regional specimen, the Kruskal-Wallis test was performed, followed by multiple comparison testing using the Steel-Dwass test with the level of significance set to 0.05. As previously, the Bonferroni adjustment was employed, and IBM SPSS statistics software was used. Furthermore, differences in μ-TBS \((n=32)\) among the four restoration types were analyzed with one-way ANOVA and Tukey’s HSD tests, with the same level of significance and software as above.

To estimate the bond reliability and bond durability of the four restorations, the Weibull modulus \((W_m)\) and Weibull stress value for PF10 and PF90, were statistically determined based on the μ-TBS data (with 0 MPa values recorded for ptf being excluded) and using Excel 2010 for Windows.

Fracture mode observation
After the μ-TBS measurements, the fracture modes of all beam-shaped specimens were observed under a light microscope (Measurescope MM-1200) at a magnification of \(×200\). After observation, the typical dentin-side surfaces of the debonded test specimens were observed under a field-emission scanning electron microscope (FE-SEM; S-4000, Hitachi, Tokyo, Japan) at \(×50\) and \(×500\) magnifications and with an accelerating voltage of 5.0 kV, to confirm the fracture mode components. Prior to the SEM observation of fractured mode, fractured surfaces of the beam-shaped specimens made of the flowable resin composite and the resin cement were prepared. Fabricated surface of the CAD/CAM ceramic block was also prepared. Two types of “cohesive fracture” occurred within flowable resin composite and resin cement and the intact fabricated surface of ceramic restorative were observed by SEM as an observation index.

RESULTS

IDS layer thickness
The differences in IDS layer thickness \((n=16)\) between the buccal and palatal regions and between the mesial and distal regions for the three IDS restoration types, excluding the N restoration group, are listed in Tables 2 and 3, respectively. Regardless of IDS type, the mean thickness value of the buccal IDS layer was significantly greater than that of the palatal case. However, there was no significant difference in thickness between the mesial and distal regions, regardless of IDS type. The IDS layer thickness was influenced by the IDS application, and increased significantly in the order of T<S<B, regardless of region.

Resin cement layer
Tables 4 and 5 list the differences in resin cement layer thickness \((n=16)\) between the buccal and palatal regions and between the mesial and distal regions, respectively, for the four restoration types. For both comparisons, no significant difference in mean thickness was determined for the resin cement layer, regardless of IDS type. The IDS application did affect the resin cement layer thickness, and the thickness values were in the order of \(T ≡ B ≡ S < N\). The values for the IDS restoration groups

| Table 2 | Differences in IDS layer thickness between buccal and palatal regions and among three IDS restoration types |
|---------|--------------------------------------------------------------------------------------------------|
| Region  | Buccal (S.D.)                                                                                   | Palatal (S.D.)                              |
| T       | 203.4 (12.6) \(^{AA}\)                                                                        | 192.5 (8.2) \(^{AA}\)                      |
| S       | 610.3 (31.6) \(^{AB}\)                                                                        | 379.5 (39.5) \(^{BB}\)                    |
| B       | 1,190.4 (57.4) \(^{AC}\)                                                                       | 1,055.4 (62.1) \(^{AC}\)                  |
| N       | none                                                                                            | none                                      |

IDS: immediate dentin sealing. Values in micrometers with different letters indicate a statistically significant difference \((p<0.05)\). Lowercase letters correspond to rows and differences between buccal and palatal regions; Uppercase letters correspond to columns and differences between the T, S, and B restorations.

| Table 3 | Differences in IDS layer thickness between mesial and distal regions and among three IDS restoration types |
|---------|--------------------------------------------------------------------------------------------------|
| Region  | Mesial (S.D.)                                                                                   | Distal (S.D.)                              |
| T       | 198.4 (11.2) \(^{AA}\)                                                                        | 197.5 (12.8) \(^{AA}\)                    |
| S       | 496.4 (127.7) \(^{AB}\)                                                                        | 493.4 (121.1) \(^{AB}\)                  |
| B       | 1,128.0 (92.8) \(^{AC}\)                                                                       | 1,117.8 (90.1) \(^{AC}\)                 |
| N       | none                                                                                            | none                                      |

IDS: immediate dentin sealing. Values in micrometers with different letters indicate a statistically significant difference \((p<0.05)\). Lowercase letters correspond to rows and differences between mesial and distal regions; Uppercase letters correspond to columns and differences between the T, S, and B restorations.
Table 4 Differences in resin cement layer thickness between buccal and palatal regions and among four restoration types

| Region | Buccal (S.D.) | Palatal (S.D.) |
|--------|--------------|---------------|
| T      | 153.3 (16.2)  | 154.2 (17.1)  |
| S      | 159.7 (19.2)  | 156.7 (18.1)  |
| B      | 156.7 (19.5)  | 155.4 (20.0)  |
| N      | 196.9 (21.0)  | 195.2 (18.6)  |

Values in micrometers with different letters indicate a statistically significant difference (p<0.05). Lowercase letters correspond to rows and differences between buccal and palatal regions; Uppercase letters correspond to columns and differences among the four restoration types.

Table 5 Differences in resin cement layer thickness between mesial and distal regions and among four restoration types

| Region | Mesial (S.D.) | Distal (S.D.) |
|--------|--------------|---------------|
| T      | 155.3 (17.8)  | 152.2 (15.3)  |
| S      | 157.2 (17.6)  | 158.7 (19.8)  |
| B      | 157.2 (19.4)  | 154.9 (20.0)  |
| N      | 197.8 (19.1)  | 194.3 (20.4)  |

Values in micrometers with different letters indicate a statistically significant difference (p<0.05). Lowercase letters correspond to rows and differences between mesial and distal regions; Uppercase letters correspond to columns and differences among the four restoration types.

Table 6 Differences in micro-tensile bond strength between buccal and palatal regions and among four restoration types

| Region | Buccal | Palatal |
|--------|--------|---------|
|        | Mean (S.D.) | Median | Max/Min | Q1 | Q3 | ptf | Mean (S.D.) | Median | Max/Min | Q1 | Q3 | ptf |
| T      | 6.3 (2.1)  | 6.3 A  | 9.8/0.0 | 5.5 | 7.3 | 1   | 5.4 (2.4)  | 5.7 A  | 8.2/0.0 | 4.8 | 7.1 | 2   |
| S      | 12.1 (2.5) | 11.6 B | 15.9/8.4 | 9.9 | 14.5 | 0   | 11.0 (1.5) | 10.6 B | 14.1/8.2 | 10.1 | 12.1 | 0   |
| B      | 12.6 (2.0) | 13.2 B | 16.0/10.1 | 10.4 | 14.2 | 0   | 11.5 (2.2) | 12.0 B | 14.1/4.8 | 10.6 | 13.1 | 0   |
| N      | 4.6 (1.1)  | 4.4 A  | 6.4/2.8 | 3.9 | 5.5 | 0   | 4.3 (1.8)  | 5.0 A  | 6.7/0.0 | 4.0 | 5.2 | 2   |

Values in MPa with different letters indicate a statistically significant difference (p<0.05). Lowercase letters correspond to rows and differences between buccal and palatal regions; Uppercase letters correspond to columns and differences among four restoration types. S.D.: standard deviation; Q1: first quartile; Q3: third quartile; ptf: number of pre-testing failure specimens.

(i.e., T, B, and S) were significantly smaller than that for the N group (without IDS application), regardless of region.

**Difference in μ-TBS between two regional specimens and among four restoration types**

The differences in μ-TBS between the buccal and palatal regions (mesial and distal regions) and among the four restoration types are presented in Table 6 and Fig. 2-[a] (Table 7 and Fig. 2-[b]). No significant difference in the mean value of μ-TBS was determined for either of the comparison sets, regardless of IDS type. However, the μ-TBS value of the MODP CAD/CAM ceramic onlay restoration did vary with the IDS application, and the values for the S and B restorations were significantly greater than those of the T and N restorations, regardless of region.

**Difference in μ-TBS among four restorations**

Three test specimens of the T restoration group and two specimens of the N restoration group were debonded during trimming before the μ-TBS test (i.e., ptf occurred); however, no ptf was evident for the S and B restorations. Figure 3 shows the difference in μ-TBS among four restorations with and without IDS application. The IDS application had an effect on the μ-TBS value of the MODP CAD/CAM ceramic onlay restoration. That is, the μ-TBS values of the IDS restorations of groups T, S,
Table 7  Differences in micro-tensile bond strength between mesial and distal regions and among four restoration types

| Region | Mesial | Distal |
|--------|--------|--------|
|        | Mean (S.D.) | Median | Max/Min | Q1 | Q3 | ptf | Mean (S.D.) | Median | Max/Min | Q1 | Q3 | ptf |
| T      | 5.9 (2.7)  | 6.7    | 9.8/0.0 | 5.2 | 7.3 | 2   | 5.7 (1.8)  | 6.1    | 8.4/0.0 | 5.2 | 6.6 | 1   |
| S      | 12.3 (2.4) | 12.2   | 15.9/8.4 | 10.1 | 14.5 | 0   | 10.8 (1.7) | 10.6   | 14.1/8.2 | 9.9 | 11.2 | 0   |
| B      | 12.8 (1.2) | 12.4   | 16.0/10.2 | 11.5 | 14.2 | 0   | 11.4 (2.3) | 11.7   | 14.1/4.8 | 10.2 | 13.1 | 0   |
| N      | 4.6 (1.6)  | 5.1    | 6.7/0.0 | 3.8 | 5.6 | 1   | 4.3 (1.4)  | 4.3    | 6.0/0.0 | 4.0 | 5.1 | 1   |

Values in MPa with different letters indicate a statistically significant difference ($p<0.05$). Lowercase letters correspond to rows and differences between mesial and distal regions; Uppercase letters correspond to columns and differences among four restoration types. S.D.: standard deviation; Q1: first quartile; Q3: third quartile; ptf: number of pre-testing failure specimens.

Fig. 2  Box plot of micro-tensile bond strength for buccal and palatal specimens [a] and for mesial and distal specimens [b].

The box indicates the data spread between the 1st and 3rd quartile. The central vertical line and circle show the median and mean, respectively. Maximum and minimum values are indicated by the whiskers. < >: Number of pre-testing failure specimens; N.S.: There is no significant difference between the buccal and palatal regions and also between the mesial and distal regions for each restoration.

Table 8 presents the differences in Weibull parameters among the four restoration types; the listed parameters are the Wm, PF10 and PF90 (95% confidence intervals, lower-upper limit values), and the number of ptf specimens. The Wm values of the four restoration types were examined via a significance test based on the regression line slope, with the level of significance set to 0.05. It was found that the IDS application influenced the Wm value of the MODP CAD/CAM ceramic onlay restoration. The Wm values decreased in the order of S>T>N>B, and a significant difference in Wm was recognized between the S and B restorations only. The S restoration group exhibited the highest performance in terms of bonding reliability based on the Wm value, and B were significantly greater than the value for the N restoration group. In addition, the values for the S and B restorations were significantly greater than that for the T restoration, and there was no significant difference between the S and B restorations.

**Difference in bonding reliability among four restorations**

Table 8 presents the differences in Weibull parameters among the four restoration types; the listed parameters are the Wm, PF10 and PF90 (95% confidence intervals, lower-upper limit values), and the number of ptf specimens. The Wm values of the four restoration types were examined via a significance test based on the regression line slope, with the level of significance set to 0.05. It was found that the IDS application influenced the Wm value of the MODP CAD/CAM ceramic onlay restoration. The Wm values decreased in the order of S>T>N>B, and a significant difference in Wm was recognized between the S and B restorations only. The S restoration group exhibited the highest performance in terms of bonding reliability based on the Wm value, and B were significantly greater than the value for the N restoration group.
Table 8  Differences in Weibull parameters among four restoration types

| Restoration (n=32) | Wm  | PF10          | PF90          | ptf |
|-------------------|-----|---------------|---------------|-----|
| T                 | 6.0<sup>AB</sup> | 4.8 (4.6–4.9)<sup>A</sup> | 8.0 (7.8–8.2)<sup>AB</sup> | 3   |
| S                 | 6.3<sup>A</sup>  | 8.7 (8.3–9.0)<sup>B</sup>  | 14.1 (13.7–14.7)<sup>B</sup> | 0   |
| B                 | 5.2<sup>B</sup>  | 8.5 (8.0–9.0)<sup>B</sup>  | 15.5 (14.8–16.4)<sup>C</sup> | 0   |
| N                 | 5.6<sup>AB</sup> | 3.4 (3.4–3.5)<sup>C</sup>  | 5.9 (5.8–6.0)<sup>D</sup>  | 2   |

Values with different letters indicate a statistically significant difference (p<0.05). Upper case letters correspond to columns and differences among four restorations. Wm: Weibull modulus; PF10, PF90: Weibull stress values in MPa for 10% and 90% probability of failure (95% confidence interval); ptf: number of pre-testing failure specimens.

Table 9  Fracture mode distribution observed using light microscope

| Sealing mode | Specimen region | T       | S       | B       | N       |
|--------------|-----------------|---------|---------|---------|---------|
|              | Buc Pal         | Buc Pal | Buc Pal | Buc Pal | Buc Pal |
|              |                 |         |         |         |         |
| Ri+Cc        | Mes Dis         | 5 8 6 5 | 8 7 4 5 | 8 7 7 7 | 5 3 6 6 |
| Di+Cc        | 0 0 0 0         |         |         |         |         |
| Cc           | 3 0 2 3         |         |         |         |         |
| ptf (Fracture mode) | (Cc) (Cc) (Cc) (Di+Cc) | (Cc) (Cc) (Cc) (Di+Cc) |
|              |                 |         |         |         |         |

Buc: buccal; Pal: palatal; Mes: mesial; Dis: distal. Ri: interfacial fracture occurred at interface between restorative/resin cement; Cc: cohesive fracture occurred within resin cement; Di: interfacial fracture occurred at interface between resin cement/dentin; ptf: number of pre-testing failure specimens.

which is defined as the reliability to achieve the specific μ-TBS value of a restoration.

Difference in bonding durability among four restorations

The differences in stress values for the PF10 and PF90 levels between test groups subjected to Weibull analysis were significant when the 95% confidence intervals did not overlap (Table 8)<sup>18</sup>. Further, the IDS application affected the PF10 and PF90 values of the MODP CAD/CAM ceramic onlay restoration. From the results of the Weibull analysis (excluding values of 0 MPa recorded for cases of ptf), the stress values at PF10 of the IDS restorations with T, S, and B applications were significantly greater than that of the N restoration group. In addition, the stress values of the S and B restorations were significantly greater than that of the T restoration. Similarly, the stress values at PF90 of the T, S, and B IDS restoration groups were significantly greater than that of the N restoration group. In addition, the value for the B restoration was significantly greater than that of the S restoration.

Fracture mode distribution

The fracture mode distributions observed using a light microscope are reported in Table 9. The fractured surfaces were classified according to one of the following fracture modes: Ri: a restorative/resin cement interface; Cc: a cohesive fracture in resin cement; and Di: a resin cement/dentin interface. Most of the post-test specimens for the four restoration types exhibited a mixed fracture consisting of Ri and Cc (Figs. 4-1 and 2). For the T, S, B, and N groups, 24, 24, 29, and 20 specimens exhibited the mixed fracture, respectively. In addition, the Cc mode (Figs. 4-3 and 4) was observed on the fractured surfaces of all specimens, regardless of IDS type. Ptf occurred for three and two specimens of the T and N restoration groups, respectively. All ptf specimens of the T restoration group exhibited the Cc mode, and every fracture surface of the N-restoration ptf specimens had Di+Cc (Figs. 4-5 and 6) characteristics.

DISCUSSION

The force generated during typical mastication of food items such as biscuits, carrots, or meat is reported to be 70–150 N<sup>19,20</sup>. Therefore, the magnitude of the load set in this study (157 N) may be similar or slightly more severe than that experienced during normal mastication. Further, the masticatory rate is 60–90 cycles/min<sup>21,22</sup> and, accordingly, the rate considered in this study may be similar to that of normal mastication. In addition, the average number of masticatory cycles...
Most of the post-test specimens for the four restoration types exhibited a mixed fracture consisting of Ri and Cc, and the rate were T: 75%, S: 75%, B: 91% and N: 63%, respectively. The rate of single Cc for the four restoration types were T: 25%, S: 25%, B: 9% and N: 25%, respectively. The mixed fracture consisting of Di and Cc was observed only in N restoration, and the rate was 13%. 1 and 2 (×50 and ×500) show the mixed fracture mode consisting of interfacial fracture at interface between restorative/resin cement (Ri) and cohesive fracture within resin cement (Cc) (a test specimen of S restoration). 3 and 4 (×50 and ×500) show cohesive fracture within resin cement (a test specimen of T restoration). 5 and 6 (×50 and ×500) show the mixed fracture consisting of interfacial fracture at interface between resin cement/dentin (Di) and cohesive fracture within resin cement (a test specimen of N restoration). Ri: restorative/resin cement interface, Cc: cohesive fracture in resin cement, Di: resin cement/dentin interface.

per year is approximately $2.5 \times 10^5$. Therefore, the cyclic loading number using in the present study is approximately equivalent to that for normal mastication over 14 months. However, the loading in this study was implemented as a condition corresponding to continuous mastication without sleep or rest.

The thickness of the IDS layer in the buccal region is clearly significantly larger than that in the palatal region, because the standardized MODP onlay cavity in this study contains only the vertical buccal wall including a part of the occlusal margin. In addition, the IDS material, a flowable resin composite, has low viscosity. The results of this study indicate that the IDS layer thickness is constant when the axial wall is always present, because no significant difference in thickness was found between the mesial and distal regions. Moreover, the results also indicate that the IDS application affects the resin cement layer thickness, as the values for the IDS restorations are significantly smaller than those of the restoration without the IDS application, regardless of region. In general, rounded diamond burs were used for the preparation of ceramic restoration, and the same diamond bur selection was done in this study. Previously, Hayashi et al. prepared a standardized cavity using a similar rounded diamond bur, and reported that the cavity adaptation of CAD/CAM restoration is improved by IDS application. The IDS application may smoothen the intra-cavity surfaces and round the angles. In that case, even a thin IDS application would improve the scanning accuracy and yield lower resin cement thickness, while also allowing better cavity adaptation than that of a restoration without IDS application.

No study has examined the effect of IDS layer thickness on the intra-cavity $\mu$-TBS value of metal-free restoration. In this study, the $\mu$-TBS values of S restorations having moderate IDS thickness (380–610 μm) and those of B restorations having a thick IDS layer (1,060–1,190 μm) were significantly greater than the values for T restorations having a thin IDS layer (150–200 μm), regardless of region. In addition, the $\mu$-TBS values of restorations with IDS applications were
significantly greater than those for restorations without IDS. Therefore, the present study clarifies that the μ-TBS value varies with IDS layer thickness, and a moderate or thick IDS layer can yield a more desirable intra-cavity bond strength compared with a thin or nonexistent IDS layer. It may be concluded that a moderate or thicker IDS layer acts as a significant stress-breaker to yield desirable intra-cavity bond strength under cyclic load stress. In this study, the mean thickness value of the buccal IDS layer was found to be significantly greater than that of the palatal value, regardless of IDS type. However, the differences between the two values for the T, S, and B applications were approximately 10, 230, and 130 μm, respectively, and significant differences in μ-TBS between the two regions were not observed. On the other hand, the differences in IDS thickness for the T, S, and B applications between the mesial and distal regions were 1, 3, and 11 μm, respectively, and no significant difference in IDS thickness or μ-TBS value between the two regions was confirmed. Therefore, under the condition that the same IDS status is applied and the difference in the IDS layer thickness is 230 μm or less, it appears that the intra-cavity μ-TBS of the CAD/CAM onlay restoration indicates a uniform value and is not influenced by the IDS application.

No study has investigated the effect of the resin cement layer thickness on the intra-cavity μ-TBS of the metal-free restoration. In this study, no significant differences in resin cement layer thickness or μ-TBS were found between the results for the buccal and palatal regions, or between those for the mesial and distal regions, regardless of IDS application. Therefore, if the same IDS application is implemented, the thickness of the intra-cavity resin cement layer for the CAD/CAM onlay restoration is uniform and the μ-TBS does not vary with cement thickness. In addition, this study found that the μ-TBS values (11.5–12.1 MPa) of the S and B restorations (380–1,190 μm IDS thickness, 150–160 μm cement thickness) was significantly greater than the value (4.4 MPa) of the N restoration (zero IDS thickness, 190–200 μm cement thickness). Therefore, it appears that the IDS thickness is a more important factor than the cement thickness as regards the μ-TBS.

In this study, the μ-TBS values of the IDS restorations with T, S, and B applications were significantly greater than the N restoration value. Previously, Feitosa et al.25 examined the effect of the IDS technique on the μ-TBS values of indirect composite restorations to the flaten dentin surfaces of extracted human third molars, after thermal and load cycling. Those researchers concluded that the IDS technique yields high bond strength for the restoration. Further, Ishii et al.8 have reported that IDS application improves the intra-cavity bond strength of metal-free CAD/CAM onlay restorations, which is in agreement with the findings of this study. However, no study has investigated the effect of IDS form and thickness on the intra-cavity μ-TBS of a CAD/CAM restoration. The results of this study indicate that the μ-TBS values of IDS restorations with T, S, and B applications are significantly greater than those for the N restoration group.

From the bond-strength test results, there is no doubt that fracturing occurs at the weakest or most damaged part of the test specimen. Most (T and S restorations: 75%; B restoration: 91%; N restoration: 63%) of the post-test specimens exhibited the Ri+Cc mixed failure mode, consisting of a cohesive fracture in the resin cement and an interfacial fracture at the interface between the restorative and resin cement, regardless of IDS type. In addition, the Di+Cc mixed failure mode, consisting of dentin-interfacial failure and cohesive failure in the resin cement, was observed for the N restoration group only.

The modulus of elasticity of the ceramic block (VITABLOC MARK II) used in this study was approximately 45 GPa26). According to the manufacturer, the elastic modulus of PANAVIA V5, which was used as a luting resin cement in the present study, is 6.3 GPa27, and the value for the Filtek Supreme Ultra Flowable Restorative used for IDS in this study is 6.8 GPa28). Further, the modulus of dentin is 16–18 GPa29,30). Based on the above, the elastic modulus values of the materials used in this study decrease in the following order: ceramic block>dentin>flowable resin composite ≒ resin cement. The mechanical properties of those materials were similarly examined, and the flexural strength values were found to increase in the following order: flowable resin composite (120 MPa)28,39 ≒ resin cement (127 MPa)37 ≒ ceramic block (154 MPa)26<dentin (213 MPa)29. The difference in elasticity across interfaces generates a high interfacial stresses, and also remarkably changes stress distributions elsewhere in the structure31. The flexibility of materials existed in cavity against the external force in this study, which acts as a stress-breaker, may increase in the following order, based on the modulus of elasticity values: ceramic block (45 GPa)26<dentin (16–18 GPa)30,31<flowable resin composite (6.8 GPa)37 ≒ resin cement (6.3 GPa)27. In addition, the fatigue resistance of the materials may increase in the following order, based on the flexural strength values: flowable resin composite (120 MPa)28<dentin (127 MPa)37<ceramic block (154 MPa)26<dentin (213 MPa)29.

The Ri+Cc fracture mode was observed in most of the post-test specimens, regardless of IDS status. Note that Ri failure may be induced by the large difference in elastic modulus between the restorative and resin cement, and the low fatigue resistance of the resin cement causes Cc failure. Further, the Di+Cc fracture mode was found to occur for the N restoration group only, because of the large difference in elastic modulus between the dentin and resin cement (due to the absence of an IDS layer), along with the low fatigue resistance of the resin cement.

A Weibull analysis is a statistical method widely applied in the field of reliability engineering, as it is particularly useful for estimating specimen reliability30. The main advantage of this analysis is that information regarding bonding effectiveness is obtained, along with an estimation of the outcome reliability30. Weibull
analysis is characterized by two principal parameters; the Wm (shape), used to predict the reliability of a bond, and the Weibull stress value (scale) of a failure. The latter is used to evaluate the performance of a bond at a constant percentage level (e.g., the 10, 63.2, and 90% levels)\(^{30}\).

Previously, Robin et al.\(^{30}\) reported that a high Wm is desirable for all materials, because it indicates excellent homogeneity in the flaw population and a more predictable failure behavior. Further, Inokoshi et al.\(^{32}\) have stated that a higher Wm corresponds to greater bonding reliability. However, Kawai et al.\(^{17}\) recommend that the bond reliability should be evaluated using both the Wm and the state of ptf occurrence, because the \(\mu\)-TBS data for Weibull analysis excludes 0 MPa values recorded for ptf specimens.

In this study, ptf occurred for three and two specimens of the T (9% of all T specimens) and N restoration (6% of all N specimens) groups, respectively. As mentioned above, it could be considered that the S restoration exhibits the highest performance in terms of bonding reliability, based on both the Wm value and the state of ptf occurrence, which is defined as the reliability of obtaining the specific \(\mu\)-TBS value of a restoration. In addition, as interfacial fractures did not occur at the interface between the resin cement and IDS layer, or at the interface between the IDS layer and dentin, IDS applications may contribute to excellent bonding of the CAD/CAM ceramic onlay restoration.

The ISO/TS 11405 guideline\(^{34}\) suggests that the stress values yielding 10 and 90% probability of failure (i.e., PF10 and PF90, respectively) constitute useful way to characterize the strength of a bond. These values indicate the stress necessary to cause bonding destruction at a particular level, i.e., PF10 or PF90. A higher stress value corresponds to greater bonding durability. Previously, De Munck et al.\(^{18}\) stated that PF10 may be a more important indicator than a mean value, as low values may reflect early failures in clinical scenarios, being more important than high values occurring in a few cases.

The Weibull stress values considered in this study were obtained for both PF10 and PF90 levels. The stress values at both PF10 and PF90 of the T, S, and B groups were found to be significantly greater than those of the N restoration group. In addition, the stress values of the S and B restoration groups were significantly greater than those of the T group, regardless of failure level. Hopp and Land\(^{35}\) have reported that rounded internal line angles are desirable for pressed ceramic restorations and for milled ceramic restorations. The rounded internal form is required because of the shapes of the burs milling the ceramic block, and also to achieve excellent cavity adaptation. IDS applications using a flowable resin composite naturally create a rounded line angle. In addition, the IDS layer between the resin cement and dentin acts as a stress breaker for external forces, such as those generated by mastication. In particular, thick-layered IDS applications (the S and B groups) increase both the PF10 and PF90 values, and these applications may increase the bonding durability of the CAD/CAM ceramic onlay restorations.

The restorations examined in this study had the following four interfaces: (a) between the restorative and resin cement, (b) between the resin cement and IDS layer, (c) between the IDS layer and dentin, and (d) between the resin cement and dentin. However, only two types of interfacial fracture were observed, occurring at (a) and (d). Therefore, the interfaces at (b) and (c) may achieve a good bonding state, with both materials being integrated well with each other. On the other hand, it is apparent that large differences in elastic modulus at both (a) and (d) may cause interfacial fractures of Ri and Di type. As noted above, ptf occurred for three and two specimens of the T and N restoration groups, respectively, but no ptf specimens were obtained for the S and B restorations. The thick IDS layer may act as a successful stress-breaker and robust interfacial bonding may be achieved between the dentin and cement, because of the absence of the dentinal interface fracture mode. Furthermore, ptf may have occurred for three specimens of the T restoration group (exhibiting the Cc mode) because of a reduction in the efficacy of the stress-breaker and its direct influence on the cement material. These failures may have occurred for the T restoration group because of the thin IDS layer compared to those of the S and B restorations.

**CONCLUSION**

In this study, IDS application was found to affect the resin cement layer thickness of an MODP CAD/CAM ceramic onlay restoration, within the study limitations. In addition, the cement thicknesses of three IDS restoration types, i.e., thin-layered low-viscosity restorative sealing (T group), slope-shaped sealing (S group), and base-shaped sealing (B group), were found to be significantly thinner than those of restorations without IDS application (N group), regardless of region. The intra-cavity \(\mu\)-TBS values of the three IDS restoration types were significantly greater than those for the N restoration specimens. The IDS application was also found to have an influence on the Wm value of the CAD/CAM restoration. The S restoration exhibited the highest-performance in terms of bonding reliability based on the Wm value, which is defined as the reliability required to achieve the specific \(\mu\)-TBS value for a restoration. The IDS application also affected the PF10 and PF90 values of the CAD/CAM restoration. Further, the stress values of the three IDS restorations at both the PF10 and PF90 levels were significantly greater than those of the N restoration specimens.

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CONFLICTS OF INTEREST
The authors do not have any financial interest in the companies producing the materials mentioned in the article.

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