INTRODUCTION

Ceria-stabilized tetragonal zirconia polycrystals/alumina (Ce-TZP/Al₂O₃) nanocomposites are employed in prosthodontic treatments as frameworks for crowns, fixed partial dentures, removable dentures, and implant-supported prostheses, where computer-aided design/computer-assisted manufacturing (CAD/CAM) systems are used to fabricate such prostheses. Both Ce-TZP/Al₂O₃ and yttria-stabilized zirconia (Y-TZP) are categorized as tetragonal zirconia polycrystals (TZPs). These materials possess an excellent biocompatibility and a high corrosion resistance, although compared to Y-TZP, Ce-TZP/Al₂O₃ has a higher mechanical strength and a greater fracture toughness.

Strong bonding between the Ce-TZP/Al₂O₃ framework and luting agents is necessary to ensure that fabricated prostheses can withstand the severe load and thermal conditions of oral environments. Several physical and chemical modification techniques have been investigated to improve the adhesive bonding of resins to Y-TZP, including air abrasion, silica coating, phosphate monomer application (as a primer or resin cement), chemical etching, laser exposure, and plasma treatments with oxygen, argon, or sulfur hexafluoride. Although the bond strengths of some resin cements to Ce-TZP/Al₂O₃ are lower than those to Y-TZP, in contrast to Y-TZP, limited information is available on the surface treatments that are effective for strengthening bonding to Ce-TZP/Al₂O₃. This study was designed to evaluate the effects of air abrasion and plasma treatment on the bond strength between resin and ceria-stabilized zirconia polycrystals/alumina (Ce-TZP/Al₂O₃). Ce-TZP/Al₂O₃ specimens were ground with #1000 silicon-carbide paper, air abraded with alumina, and then exposed to glow-discharge plasma. The specimens without air abrasion and/or plasma exposure were also prepared as controls. The specimens were bonded to resin composite disks with a self-adhesive resin cement or a luting composite containing no functional monomer (LC). Shear bond strengths were determined after 10,000 thermocycles at 4 and 60°C, and the data were analyzed by non-parametric tests. When using SA, the Abrasion/Plasma specimens exhibited the highest bond strength, followed by the Abrasion/No plasma, No abrasion/Plasma, and No abrasion/No plasma specimens. For LC, neither air abrasion nor plasma treatment exhibited any significant effect on bond strength.

MATERIALS AND METHODS

The substrate materials and resin cement used are summarized in Table 1. A total of 160 specimens were ground with #600 and #1000 silicon-carbide paper, air abraded with alumina, and then exposed to glow-discharge plasma. The specimens without air abrasion and/or plasma exposure were also prepared as controls. The specimens were bonded to resin composite disks with a self-adhesive resin cement (SA) or a luting composite containing no functional monomer (LC). Shear bond strengths were determined after 10,000 thermocycles at 4 and 60°C, and the data were analyzed by non-parametric tests. When using SA, the Abrasion/Plasma specimens exhibited the highest bond strength, followed by the Abrasion/No plasma, No abrasion/Plasma, and No abrasion/No plasma specimens. For LC, neither air abrasion nor plasma treatment exhibited any significant effect on bond strength.
Table 1  Substrate material and resin cement used in the present study

| Name (Abbreviation)          | Component                                                                                       | Manufacturer (Lot No.)                      |
|-----------------------------|-------------------------------------------------------------------------------------------------|---------------------------------------------|
| **Substrate materials**     |                                                                                                 |                                             |
| NANOZR Ce-TZP/Al2O3         | ZrO2 67.9, Al2O3 21.5, CeO2 10.6, MgO 0.06, TiO2 0.03 (mass%)                                  | Panasonic Health Care, Osaka, Japan          |
| Clearfil AP-X A3            | Bis-GMA, TEGDMA, glass powder, silica filler, photoinitiator, pigments                          | Kuraray Noritake Dental, Tokyo, Japan (620075) |
| **Resin cement**            |                                                                                                 |                                             |
| Clearfil SA                 | Paste A: Bis-GMA, TEGDMA, MDP, HEMA, methacrylate monomer, barium glass filler, silica filler,  | Kuraray Noritake Dental (3P0129)            |
| Luting Plus (SA)            | photoinitiator, chemical initiator, pigments                                                   |                                             |
| Luting composite of Panavia | Paste A: Bis-GMA, TEGDMA, methacrylate monomer, barium glass filler, sodium fluoride,         | Kuraray Noritake Dental (960039)            |
| V5 without primer (LC)      | polymerization initiator, pigments                                                              |                                             |
|                             | Paste B: Bis-GMA, methacrylate monomer, barium glass filler, aluminium oxide filler,            |                                             |
|                             | photoinitiator, polymerization initiator                                                         |                                             |

Bis-GMA: bisphenol-A-diglycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; MDP: 10-methacyryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate.

Fig. 1  Flow chart of the specimen preparation process and subsequent contact angle measurements and shear bond tests.

dried.

The specimens were assigned to four pretreatment groups (Abrasion/Plasma, Abrasion/No plasma, No abrasion/Plasma, and No abrasion/No plasma) each of which contained eight specimens. In the Abrasion/Plasma and No abrasion/Plasma groups, the Ce-TZP/Al2O3 specimens were irradiated for 60 s using a glow-discharge plasma apparatus (YHS-R, Sakigake Semiconductor, Kyoto, Japan), which was operated at 100 V, 5.0 A, and 60 Hz under a vacuum environment. No special gas was used for the plasma treatment.

Contact angle measurements
Thirty-two Ce-TZP/Al2O3 disks were assigned to four
pretreatment groups as described above. The contact angles of the Ce-TZP/Al₂O₃ surfaces were measured using a contact angle meter (CA-D, Kyowa Interface Science, Nii, Japan). One point eight microliters of distilled water was dropped onto the Ce-TZP/Al₂O₃ specimen. After 10 s, the contact angle, defined by the tangent line between the water droplet and specimen surface, was measured at 20°C.

Shear bond tests
One hundred and twenty-eight Ce-TZP/Al₂O₃ disks were divided into four pretreatment groups. A piece of 50 µm-thick masking tape with a circular hole (5 mm diameter) was attached to each Ce-TZP/Al₂O₃ specimen to define the thickness and the bonding area. A self-adhesive resin cement (SA; Clearfil SA Luting Plus, Kuraray Noritake Dental, Tokyo, Japan) or a luting composite (LC; Kuraray Noritake Dental) was applied to the bonding area, and a pre-polymerized resin composite disk (Clearfil AP-X A3, Kuraray Noritake Dental, 8 mm diameter, 2 mm height) was placed at the center of the bonding area and affixed to the Ce-TZP/Al₂O₃ specimen with finger pressure. Light was then irradiated from directly above the bonded resin composite disk for 20 s using a light emitting diode (LED) light unit (Pencure, J. Morita).

The bonded specimens were allowed to stand at room temperature for 30 min, and then immersed in distilled water at 37°C for 24 h. Half of the specimens (four sets of eight) were tested for their shear bond strength values (designated as thermocycle 0). The remaining four sets were subjected to thermocycling for 10,000 cycles alternating between water baths held at 4 and 60°C, with a 1 min dwell time per bath. The specimens were embedded in an acrylic mold and fitted to a shear-testing jig (No. ISO/TR11405, Wago Ind., Nagasaki, Japan) that was used to apply a shearing load parallel to the bonded interface (Fig. 2). The shear bond strength, which is the force at failure divided by the bonded surface area, was determined using a universal testing machine (AGS-10kNG, Shimadzu, Kyoto, Japan) at a cross-head speed of 0.5 mm/min.

Failure mode observations
After shear testing, the Ce-TZP/Al₂O₃ and the resin composite disk surfaces of the debonded specimens were observed by an optical microscopy (SMZ-10, Nikon, Tokyo, Japan) at a magnification of 20× to determine the type of bond failure. The failure mode was categorized as adhesive failure at the resin cement–Ce-TZP/Al₂O₃ interface (Ad), cohesive failure within the resin cement (Co), or mixed failure through a combination of these modes (Ad/Co).

Scanning electron microscopy (SEM)
A debonded Ce-TZP/Al₂O₃ specimen was sputter-coated with gold (Ion Coater IB-3, Eiko Engineering, Hitachinaka, Japan) prior to observation by SEM (JCM-6000Plus, JEOL, Tokyo, Japan) at magnification of 500×.

Statistical analysis
The mean contact angle, bond strength, and the standard deviation (SD) of eight specimens were calculated for each test group. The homoscedasticity assumption was assessed by Levene tests, and the data were analyzed by Steel-Dwass and Wilcoxon tests at a statistical significance of 0.05. Statistical analysis was carried out using the JMP Pro software system (SAS Institute Japan, Tokyo, Japan).

RESULTS

Contact angle
The mean contact angle was found to vary from 1.4 to 60.0° (Table 2). The smallest contact angle was obtained for the Abrasion/Plasma specimens, and the values for the different groups increased according to the following order: No abrasion/Plasma, Abrasion/No plasma, and Abrasion/Plasma.

Shear bond strength
The mean bond strength varied from 2.3 to 30.0 MPa (Tables 3 and 4). When SA was employed, the No abrasion/Plasma, Abrasion/No plasma, and Abrasion/Plasma specimens exhibited higher bond strengths than the No abrasion/Plasma specimen at thermocycle 0 (Table 3). After 10,000 thermocycles, the Abrasion/Plasma specimens exhibited the highest bond strength, followed by the Abrasion/No plasma and No abrasion/Plasma specimens, while the No abrasion/No plasma specimens showed the lowest bond strength. No significant differences were found between the No abrasion/Plasma and Abrasion/No plasma specimens, or between the Abrasion/No plasma and Abrasion/Plasma specimens.

When LC was employed, the bond strengths of the Abrasion/No plasma and Abrasion/Plasma specimens were significantly higher at thermocycle 0 than that of the No abrasion/No plasma specimen; however, the difference between the No abrasion/Plasma and No abrasion/No plasma specimens was not statistically significant (Table 4). After 10,000 thermocycles using LC, no significant differences were observed among the four groups.
Table 2  Means and standard deviations (SDs) of contact angle

| Group                  | Contact angle (degree) | Mean* | SD  |
|------------------------|------------------------|-------|-----|
| No abrasion/No plasma  |                         | 60.0a | 1.1 |
| No abrasion/Plasma     |                         | 4.2b  | 1.1 |
| Abrasion/No plasma     |                         | 10.8c | 0.9 |
| Abrasion/Plasma        |                         | 1.4d  | 0.4 |

*Identical superscript letters indicate that the values are not statistically different (Steel-Dwass test, p>0.05).

Table 3 Shear bond strength between Ce-TZP/Al₂O₃ and SA, and failure mode

| Group name                               | Shear bond strength (MPa) | Failure modes** (number of specimens) |
|------------------------------------------|--------------------------|---------------------------------------|
|                                          | Thermocycle 0            | Thermocycle 10,000                    | Thermocycle 0 | Thermocycle 10,000 |
|                                          | Mean* | SD   | Mean* | SD   | Ad/Co(8) | Ad(4) | Ad/Co(4) | Ad/Co(8) |
| No abrasion/No plasma                    | 16.0bA 3.5 | 11.9bB 1.9 | Ad/Co(8) | Ad(4) | Ad/Co(4) |
| No abrasion/Plasma                       | 24.6bA 3.7 | 16.3bB 2.6 | Ad/Co(8) | Ad/Co(8) |
| Abrasion/No plasma                       | 30.0bA 3.4 | 22.1bB 5.7 | Ad/Co(8) | Ad/Co(8) |
| Abrasion/Plasma                          | 26.2bA 4.1 | 28.4bA 3.8 | Ad/Co(8) | Ad/Co(8) |

*Means with the same small letters (grouped by Steel-Dwass test) in the same columns or capitals (grouped by Wilcoxon test) on each horizontal line were not significantly different (p>0.05).

**Ad: adhesive failure at the interface between Ce-TZP/Al₂O₃ specimen and resin cement; Co: cohesive failure within resin cement; Ad/Co: mixed failure of Ad and Co.

Table 4 Shear bond strength between Ce-TZP/Al₂O₃ and LC, and failure mode

| Group name                               | Shear bond strength (MPa) | Failure modes** (number of specimens) |
|------------------------------------------|--------------------------|---------------------------------------|
|                                          | Thermocycle 0            | Thermocycle 10,000                    | Thermocycle 0 | Thermocycle 10,000 |
|                                          | Mean* | SD   | Mean* | SD   | Ad(8) | Ad(8) |
| No abrasion/No plasma                    | 3.7xa | 1.2  | 2.3xa | 0.6  | Ad(8) | Ad(8) |
| No abrasion/Plasma                       | 4.9xbA | 2.2  | 2.4xb | 0.2  | Ad(8) | Ad(8) |
| Abrasion/No plasma                       | 7.3xcA | 1.6  | 2.3xcA | 0.4 | Ad(8) | Ad(8) |
| Abrasion/Plasma                          | 9.2xA | 3.0  | 2.4xA | 0.6  | Ad(8) | Ad(8) |

*Means with the same small letters (grouped by Steel-Dwass test) in the same columns or capitals (grouped by Wilcoxon test) on each horizontal line were not significantly different (p>0.05).

**Ad: adhesive failure at the interface between Ce-TZP/Al₂O₃ specimen and resin cement.

Failure mode

For all groups at thermocycle 0, the specimens bonded with SA exhibited Ad/Co failure (Table 3). After 10,000 thermocycles, with the exception of four No abrasion/No plasma specimens that exhibited Ad failure, all specimens bonded with SA failed in the Ad/Co mode. In contrast, all the specimens bonded with LC exhibited Ad failure both before and after thermocycling (Table 4). No failure occurred at the interface between the resin cement and the resin composite substrate material, and neither complete cohesive failure within the resin cement nor fracture within the substrate materials was observed in any specimen.

SEM

Figure 3 shows the Abrasion/Plasma specimen, which was bonded with SA and failed in the Ad/Co mode at thermocycle 0. More specifically, the scanning electron micrograph of the debonded specimen reveals that traces of fractured resin cement remained on the air-abraded Ce-TZP/Al₂O₃ surface.
DISCUSSION

The present study revealed that the bond strength of Ce-TZP/Al₂O₃ after thermocycling was significantly improved by surface treatment with glow-discharge plasma and air abrasion, and so the null hypothesis was rejected.

The experimental conditions employed herein for air abrasion were determined based on previous studies that reported the following parameters for Y-TZP or Ce-TZP/Al₂O₃: air pressure (0.05–0.45 MPa), distance from the nozzle to the specimen (5–20 mm), and duration (5–30 s)⁵,¹⁴,³¹. It is known that air abrasion using alumina can roughen Ce-TZP/Al₂O₃ surfaces, thereby increasing the actual bonding area³¹ and decreasing the amount of organic contaminants on the substrate surface.⁵⁴ Although the wettability of the No abrasion/Plasma specimens may be higher than that of the Abrasion/No plasma specimens, the bond strength of the former was not superior to that of the latter. Such a discrepancy suggests that wettability is a necessary condition to achieve a high bond strength, but does not always indicate high bond strength, as bond strengths are determined by multiple factors. We therefore considered that the air-abraded Ce-TZP/Al₂O₃ specimens may generate larger friction forces due to the rough surface during shear bond testing.

In addition, plasma is known to promote the formation of active peroxide radicals (R-O-O·), which generate oxygen-containing functional groups (-O and -OH) on the plasma-exposed surface.²⁰ Indeed, X-ray photoelectron spectroscopy (XPS) has revealed that plasma treatment increases the number of oxygen functional groups and decreases the quantity of carbon on Y-TZP surfaces.²⁴,²⁹ Therefore, the plasma treatment may promote the formation of the oxygen functional groups on the Ce-TZP/Al₂O₃ specimens.

Several studies have indicated that the use of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) is advantageous to zirconia bonding.¹⁷-²⁰ Nuclear magnetic resonance (NMR) studies have revealed that ionic and hydrogen bonds can be created between MDP and zirconia, while Chuang et al.³⁹ reported the occurrence of MDP-O-Zr covalent bonds on the Y-TZP surfaces using time-of-flight secondary ion mass spectrometry. The chemical bonding during time to MDP and Y-TZP has also been examined by thermogravimetric analysis, inductively coupled plasma-mass spectroscopy, XPS, and Fourier-transform infrared spectroscopy.²⁰ The Ce-TZP/Al₂O₃ specimen possesses zirconia as a main component as well as Y-TZP.¹⁰,¹² As such, we herein selected an MDP-containing resin cement in anticipation of the chemical bonding between MDP and Ce-TZP/Al₂O₃.

In terms of the contact angle measurements, we note that the contact angle (60.0°) of the No abrasion/No plasma group measured on Ce-TZP/Al₂O₃ was close to that measured on polished Y-TZP (i.e., 51.7°).³⁰ The contact angle is a fundamental parameter to indicate the wettability of a surface, and a strong correlation exists between the contact angle and the bond strength in the case of Y-TZP.²⁰ The present findings therefore indicate that plasma treatment improves the wettability of Ce-TZP/Al₂O₃, and this result is consistent with many reports regarding Y-TZP.²⁵-³⁰

Furthermore, thermocycling and shear bond strength are both well-documented in vitro tests for determining the durability of bonding to assess the reliability of commercially available or experimental adhesive systems.¹³,¹⁵,¹⁷,¹⁸,¹⁹,²¹-²³,²⁵-²⁷,²⁹-³² In this context, several studies have investigated the bond strengths between Y-TZP and resin-based materials using thermocycling.¹³,¹⁷,³¹,³² In addition, thermal stress was found to induce the expansion and contraction of the substrate materials, accelerating the diffusion of water into the bonded interface, and 10,000 thermocycles were correlate to approximately one year in vivo.³⁰

With regards to the failure mode, half of the No abrasion/No plasma specimens exhibited Ad failure after 10,000 thermocycles (Table 3). When compared to the No abrasion/No plasma group, the absence of Ad failure in the No abrasion/Plasma group indicated that plasma treatment contributed to adhesive bonding.

In the present study, the dual-curing cements were irradiated with light through the resin composite specimens. We assumed clinical situations including the bonding of resin composite crowns to frameworks composed of Ce-TZP/Al₂O₃. In addition, Alovissi et al. reported that bond strength of a dual-curing cement (Panavia SA cement, Kuraray Noritake Dental) joined between zirconia and resin composite specimens was not affected by variation in the light-curing time (no irradiation, 20 s, or 120 s) when light curing was
performed by placing the light source on the opposite side of the zirconia surface\(^7\). It suggests that chemical cure is of particular importance for adhesion when light cannot penetrate enough through restorations.

According to the user instructions for Panavia V5, the application of an MDP-containing primer (e.g., Clearfil Ceramic Primer Plus, Kuraray Noritake Dental) onto zirconia is recommended by the manufacturer. To eliminate the performance of the functional monomer, MDP, the luting composite (LC) containing no functional monomer, was used as a control. The present findings suggest that no strong bonding is achieved without the functional monomer. Therefore, clinicians should follow the manufacturer’s instructions when using Panavia V5, and we note that further studies are required to reveal the additional effects of primers and adhesives containing different types of functional monomers.

Moreover, the results suggest that glow-discharge plasma treatment and air abrasion appear to be useful in improving the bond strengths between Ce-TZP/Al\(_2\)O\(_3\)-based superstructures and implant-abutment components or between Ce-TZP/Al\(_2\)O\(_3\)-based frameworks and resin composite crowns. Although further research is required, the combination of air abrasion and plasma treatment may contribute to enhancing the stability of resin-bonded prostheses, such as resin-bonded fixed partial dentures, which are fabricated using the Ce-TZP/Al\(_2\)O\(_3\) materials.

**CONCLUSIONS**

Within the limits of the present study, the following conclusions can be drawn:

1. The contact angle of Ce-TZP/Al\(_2\)O\(_3\) was significantly reduced following both air abrasion and glow-discharge plasma treatment alone, but was further reduced upon combination of the two treatment methods.

2. The shear bond strength between Ce-TZP/Al\(_2\)O\(_3\) and an MDP-containing resin cement was significantly enhanced following glow-discharge plasma treatment along with air abrasion.

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