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## Original article

# Influence of atmospheric pressure low-temperature plasma treatment on the shear bond strength between zirconia and resin cement

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## ARTICLE INFO

## Article history:

Received 28 July 2015  
Received in revised form  
6 January 2016  
Accepted 8 February 2016  
Available online xxx

## Keywords:

Atmospheric-pressure low-temperature plasma treatment  
Adhesive resin cement  
Zirconia  
Alumina sandblast treatment

## ABSTRACT

**Purpose:** Zirconia exhibits excellent strength and high biocompatibility in technological applications and it has therefore been investigated for clinical applications and research. Before setting prostheses, a crown prosthesis inner surface is sandblasted with alumina to remove contaminants and form small cavities. This alumina sandblasting causes stress-induced phase transition of zirconia.

Atmospheric-pressure low-temperature plasma has been applied in the dental industry, particularly for adhesives, as a surface treatment to activate the surface energy and remove contaminants.

The purpose of this study was to examine the influence of atmospheric-pressure low-temperature plasma treatment on the shear bond strength between zirconia and adhesive resin cement.

**Methods:** The surface treatment method was classified into three groups: untreated (Cont group), alumina sandblast treatment (Sb group), and atmospheric-pressure low-temperature plasma treatment (Ps group).

Adhesive resin cement was applied to stainless steel and bonded to zirconia. Shear adhesion tests were performed after complete hardening of the cement. Multiple comparisons were performed using a one-way analysis of variance and the Bonferroni method.

X-ray diffractometry was used to examine the change in zirconia crystal structure.

**Results:** Statistically significant differences were noted between the control and Sb groups and between the control and Ps groups. In contrast, no statistically significant differences were noted for the Ps and Sb bond strength.

Atmospheric-pressure low-temperature plasma treatment did not affect the zirconia crystal structure.

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<http://dx.doi.org/10.1016/j.jpor.2016.02.001>

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**Conclusions:** Atmospheric-pressure low-temperature plasma treatment improves the bonding strength of adhesive resin cement as effectively as alumina sandblasting, and does not alter the zirconia crystal structure.

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## 1. Introduction

Zirconia is a material with excellent physical and technological characteristics and high biocompatibility and it has been investigated in clinical research and applications as a frame material for crowns and fixed partial dentures [1–3]. The application of all-ceramic restorations using zirconia has spread rapidly in clinical situations. Crown restoration materials have occasionally been sold commercially. In many cases, conventional methods have been implemented for each material with slight modifications for surface treatment before setting prostheses. For example, alumina sandblasting before setting crown prostheses has been reported to cause a stress-induced phase transition in zirconia from a tetragonal to monoclinic structure [4]. A decrease in bonding strength and a change in the coefficient of expansion have been reported with the transition in zirconia crystal structure [5]. Therefore, research into an appropriate surface treatment method for zirconia as a metal-substituting material is indispensable. In addition, deformation of the margin of the prosthesis and chipping in alumina sandblasting treatment has also been reported [6].

Recently, atmospheric-pressure low-temperature plasma treatment has been applied in the dental industry, particularly for adhesives, as a surface treatment that can activate the surface energy and remove contaminants [7]. Because atmospheric-pressure low-temperature plasma treatment carried out on a molecular level, it is possible to modify only the surface layer without inhibiting the bulk performance of the adherent. Furthermore, instead of using a wet process with an organic solvent, this method represents a dry treatment with low environmental impact. In medical fields, this method has been used to sterilize precision instruments such as medical devices and hollow instruments [8]. However, limited information has been reported on the application of atmospheric-pressure low-temperature plasma treatment to crown restorative materials.

The hypothesis of this study that atmospheric-pressure low-temperature plasma treatment does not damage the zirconia surface and is effective for pre-adhesive treatment.

On the basis of the above hypothesis, this study examined the effects of atmospheric-pressure low-temperature plasma processing on zirconia with regards to bond strength to adhesive resin cement and zirconia crystal structure.

## 2. Materials and methods

### 2.1. Study design

In this study 27 zirconia specimens classified into the three groups ( $n = 9$ ) according to the surface treatment conditions;

the untreated group (Cont group), the alumina sandblast treatment group (Sb group), and the atmospheric-pressure low-temperature plasma treatment group (Ps group). The effects of atmospheric-pressure low-temperature plasma treatment on zirconia were evaluated using shear adhesion tests and X-ray diffractometry (XRD).

A list of materials and devices that were used is provided in Tables 1 and 2.

Zirconia was processed in sections (11 mm × 11 mm × 3 mm), polished to No. 600 with a waterproof abrasive paper (Taisui Paper, Nihonkensi, Osaka, Japan) and each section was cleaned ultrasonically with acetone and distilled water for 15 min. The three conditions for surface treatment were: untreated (cont group), alumina sandblasting treatment (Sb group), and atmospheric-pressure low-temperature plasma treatment (Ps group).

Atmospheric-pressure low-temperature plasma processing was performed from an irradiation distance of 10 mm for 30 s. In the alumina sandblasting treatment, 50- $\mu$ m alumina was used from an injection distance of 30 mm, with an injection time of 10 s and an injection pressure of 0.3 MPa. Nine samples were prepared under each set of conditions. Helium was used as the active gas in the atmospheric-pressure low-temperature plasma processing (Fig. 1A and B) (Helium gas, Taiyo Nippon Sanso, Tokyo, Japan). After surface treatment, adhesive resin cement was applied to stainless steel (6 mm diameter, 5 mm high, SUS303, Stainless Syouji, Tokyo, Japan) to be bonded to the zirconia. Stainless steel specimens that bond strongly with adhesive resin cement were used to evaluate adhesion testing by the same method used in other studies [9]. Irradiation was performed from four directions for 3 s with a halogen light-curing unit (Hyper Lightel, Kuraray Medical, Tokyo, Japan) under a 15-kg load using a constant-load device. Excess cement was removed before the sample was subjected to light irradiation for 20 s.

After complete curing of the cement, samples were embedded in an autopolymerizing resin (Unifast III, GC, Tokyo, Japan), and specimens were immersed in water at 37 °C for 24 h for measurement. Bonding strength was measured using a universal testing machine (Autograph AGSJ-5kN, Shimadzu, Kyoto, Japan) by setting the crosshead speed to 0.5 mm/min to perform the shear adhesion tests (Fig. 2). The maximum measuring load value was used as the cement bonding strength.

Statistical analysis was performed by a one-way analysis of variance with surface treatment as a factor. When a statistically significant difference was noted, the Bonferroni method was used to perform multiple comparison tests. All analyses were performed using IBM SPSS Statistics, Version 19 (IBM Corp., Armonk, NY, USA). The level of significance was set to 1%. Power analysis was performed for support the sample size. Power ( $1 - \beta$ ) was calculated from the sample size, the level

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