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Effect of different surface treatments on bond strength, surface and microscopic structure of zirconia ceramic

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A B S T R A C T

Objectives: To evaluate the effect of different surface treatments; plasma treatment, silica coating using plasma technology and sandblasting, on bond strength, surface roughness and microscopic structure of yttria-stabilized tetragonal zirconia polycrystals(Y-TZP) after thermo-cycling.
Matrials and methods: One hundred discs (n = 100) of yttria-stabilized tetragonal zirconia were prepared from (Y-TZP) ceramic blocks using MAD/MAM milling technology, and were divided into four equal groups (n = 25) according to the type of surface treatment. Group (1): control (no surface treatment). Group (2): zirconia discs were sandblasted by alumina particles. Group (3): zirconia discs treated by plasma technology to produce surface roughness. Group (4): zirconia discs coated by silica using plasma technology. Samples of each group were subdivided into four subgroups according to different analytical techniques. Subgroup (A): (n = 10) subjected to testing of bond strength of zirconia discs to adhesive resin cement after thermo-cycling. Subgroup (B): (n = 5), to evaluate the microscopic changes of zirconia discs by scanning electron microscope (SEM). Subgroup(C): (n = 5) to evaluate the crystal structure and phase transformation of YZ ceramic by X-ray diffraction (XRD). Subgroup (D): (n = 5) to measure three dimensional surface roughness of YZ ceramic by optical interference microscope.
Results: Statistical analysis of shear bond strength by ANOVA revealed the presence of no statistically significant difference between group (3) and (4); both showed the statistically significantly highest mean shear bond strength values. Group (2) showed statistically significantly lower mean values followed by group (1). SEM showed that the topographic pattern differed by different surface treatments of samples. XRD revealed that; group (1) showed the statistically significantly highest mean % of zirconium oxide (Tetragonal phase). Group (2) showed the statistically significantly lowest mean % of Zirconium oxide (Tetragonal phase) and highest mean % of Boehmite and Zirconium oxide (Anorthic phase); Group (3) and (4) showed the statistically significantly highest mean % of Zirconium oxide (Monoclinic phase) and low % of zirconium oxide (Tetragonal phase). 3D- optical surface roughness showed that group (3) and (4) had highest mean (Ra) values. Group (2) showed statistically significantly lower mean values. Group (1) showed the statistically significantly lowest mean (Ra) values.
Conclusions: (1) Surface treatments of Y-TZP ceramic together with MDP primer and silane-coupling agent application improve the bond strength to resin cement. (2) Plasma-Silica coating and plasma-oxygen treatment, both are valuable methods that improve the bond strength of resin cement to Y-TZP ceramic. (3) Silica coating by plasma technology provides durable bond strength and can be a promising alternative pretreatment before silane application to enhance bonding with zirconia ceramic. (4) Tetragonal-monoclinic phase transformation had occurred in Y-TZP samples received both types of plasma treatment.

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1. Introduction

New ceramic systems have been developed as attempts to eliminate metal infrastructures and allow optimal distribution of reflected light, providing high quality aesthetic restorations through the use of reinforced ceramic cores either by dispersion of leucite [1], glass infiltration into sintered alumina (Al_2O_3) [2,3], the use of high-purity alumina [4] or zirconium dioxide (zirconia, ZrO_2) [5]. Zirconia has emerged as a versatile and promising ceramic material because of its biological, mechanical and optical properties. With a flexural strength of more than 900 MPa, fracture toughness of up to 10 MPa/$m^{0.5}$, and an elastic modulus of 210 GPa, they exhibit better mechanical performance, superior strength and fracture resistance than do other ceramic materials [5,6]. Zirconium dioxide as a dental material has a wide range of applications [7], they were initially used for endodontic dowels and implant abutments [8,9]. Its use has been extended to single crowns [10,11] and posterior three-unit fixed partial dentures which can be fabricated with a manual copy-milling machine, or computer aided design/ computer aided manufacturing (CAD/CAM) systems [12,13] of either pre-sintered [14] or fully sintered zirconia blocks [15].

Adhesion of resin cement to high-strength zirconia ceramics is not expected to be improved by acid etching and silanization because they are inert acid resistant ceramics [16–21]. For zirconia ceramics, airborne-particle abrasion is an alternative method for roughening the ceramic surface [20,22,23].

However, there are some possibilities for improving bonding to zirconia based ceramics that need to be tested, including modern techniques for surface treatments by plasma technology. Plasma is defined as a gas in which part of the particles that make up the matter are present in ionized form. This is achieved by heating the gas leading to dissociation of the molecular bonds and subsequently ionization of the free atoms. Thereby, plasma consists of positively and negatively charged ions and negatively charged electrons as well as radicals, neutral and excited atoms and molecules [24,25].

In material science, possible applications of plasma include the modification of surface properties like electrochemical charge or amount of oxidation, wettability, hardness, resistance to chemical corrosion, the water absorption capacity as well as the affinity toward specific molecules can be modulated [26], using the common plasma gas sources as oxygen, argon nitrogen or hydrogen.

Some studies have examined the effect of sandblasting on shear bond strength of resin cement to zirconia ceramic, but further data is needed to correlate the effect of sandblasting and different plasma modalities on shear bond strength, microstructure, surface roughness and phase transformation of yttria-stabilized tetragonal zirconia.

2. Materials and methods

To conduct the present study, one hundred discs ($n = 100$) of Yttria-stabilized tetragonal zirconia ceramic (Y-TZP) were fabricated using Manual-aided Design/Manual-aided Manufacturing (MAD/MAM) system (Talent dental, FP50-XP, Korea) referred to as copy milling technique which is based on the pantographic principle.

2.1. Preparation of composite resin pattern

In order to standardize the shape and dimensions of the samples, a specially designed Teflon mold was machine-cut in order to fabricate circular resin discs of 10 mm diameter and 3 mm thickness. The inner walls of the mold were painted with separating medium (*Vasline petroleum jelly, Mainland, China*) then composite resin (*Te-Econom Plus, Ivoclar Vivadent, Liechtenstein*) layers were incrementally condensed into the mold and light cured (*XL- 3000, 3M/ESPE, St. Paul, USA*) for 40 s on each side, for a total of 120 s. After complete polymerization, the composite resin pattern was removed from mold and inspected for any deficiencies which if found were corrected by addition.

2.2. Milling of Y-TZP samples

The composite resin pattern was placed in the pantographic machine. The copying arm of the machine traced the composite pattern while the cutting arm, which had a carbide cutter (*TF14, Syandent tools, China*) milled the pre-sintered zirconia block. After completion of the milling process, the milled discs were separated and handled with care to avoid damage to their margins or initiation of microscopic cracks leading to subsequent failure.

2.3. Sintering process of zirconia discs

The milled zirconia discs were sintered in high temperature furnace (*Wholesale Sintering Furnace, Ds-1700MX, Mainland, China*) according to the manufacturer's recommendations. The temperature was raised to 1500 °C in 2 h then kept at final temperature (1500 °C) for 2 h. Samples were slowly cooled to less than 100 °C in 1 h.

2.4. Surface treatments of samples

One hundred discs ($n = 100$) of Yttria-stabilized tetragonal zirconia ceramic (Y-TZP) were divided into four equal groups, ($n = 25$ each) according to type of surface treatment; Group (1): control (no surface treatment). Group (2): zirconia discs were sandblasted by alumina particles. Group (3): zirconia discs treated by plasma technology to produce surface roughness. Group (4): zirconia discs coated by silica using plasma technology.

2.4.1. Sandblasting of the samples, group (2)

1. Each sample was individually mounted in a specially constructed holder which aided in standardization of the distance of sample exposure from the sandblasting nozzle (10 mm).
2. The sample was sandblasted with 110 μm aluminum oxide, at 2 bar pressure, for 15 s [27], using sandblasting machine (*Sandstorm, Vaniman manufacturing Co, Fallbrook, California, US*).
3. After sandblasting, samples were cleaned using water and air stream to remove any remnants of alumina particles on the surfaces.

2.4.2. Oxygen etching by plasma technology; group (3)

Constructed zirconia samples of group (3) were etched using plasma technology. For this purpose, oxygen gas was used as the working gas in the plasma focus system, Fig. 1, and the condenser bank was charged to 12 kV. The substrate holder that holds the samples was incorporated in the vacuum chamber facing the rim of the anode. The capacitor bank potential was transformed to the plasma focus tube through the air spark gap, in this state the plasma focus was formed after that it broke into ions and electron beams. The energetic oxygen ion beam took the shape of fountain and spread upwards to bombard the facing samples. To enhance the treatment, the process was repeated 15 times.

2.4.3. Silica coating by plasma technology; group (4)

Constructed zirconia samples of group (4) were silica coated using plasma technology. Argon gas was used as working gas; an