Materials Science and Engineering C 46 (2015) 103–110

Contents lists available at ScienceDirect

Materials Science and Engineering C

journal homepage: www.elsevier.com/locate/msec

Silica coating of zirconia by silicon nitride hydrolysis on adhesion promotion of resin to zirconia

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ARTICLE INFO

Article history:
Received 13 August 2014
Received in revised form 19 September 2014
Accepted 8 October 2014
Available online 13 October 2014

Keywords:
Zirconia
Resin cement
Silica coating
Phase transformation
Silane coupling agent
Bond strength

ABSTRACT

In this study, the effect of silica coating on zirconia by silicon nitride hydrolysis in resin zirconia bonding was investigated. The silica coated zirconia samples were prepared in silicon nitride dispersion at 90 °C under different immersion times followed by a thermal treatment at 1400 °C. Four test groups were prepared: 1) zirconia samples treated by sandblasting, 2) zirconia samples treated by immersion in silicon nitride dispersion for 6 h, 3) zirconia samples treated by immersion in silicon nitride dispersion for 24 h and 4) zirconia samples treated by immersion in silicon nitride dispersion for 48 h. The coatings were characterized by SEM, EDX, XRD and Raman. The resin zirconia bond strengths of the four test groups were evaluated under three storage conditions: dry storage, water storage in deionized water at 37 °C for 30 days and thermo-cycling for 6000 cycles between 5.0 and 55.0 °C. Surface morphology and composition of zirconia were changed after surface treatments. Phase transformation was observed for zirconia surface by sandblasting treatment but was not observed for zirconia surface treated with silicon nitride hydrolysis. Significant differences in bond strengths were found under different surface treatments ($p < 0.001$) and under three storage conditions ($p < 0.005$). The highest bond strength values were obtained by sandblasting treatment.

1. Introduction

Zirconia is a polymorphic material that can exist in three allotropic forms: monoclinic, tetragonal and cubic. The tetragonal phase is stable at high temperature but can be stabilized at room temperature by adding 3 mol% yttria (Y_2O_3) [1]. Yttria-stabilized tetragonal zirconia (referred as "zirconia" in the following text) is an important biomaterial used in dentistry because of its excellent biocompatibility and mechanical properties [2]. However, zirconia is chemically inert and it is difficult to attain strong bonding to it with other organic materials such as dental resin cements. A surface treatment is one of the approaches to improve the resin zirconia bonding. The physical and chemical properties of zirconia surface are changed after surface treatments in such a way that the modified zirconia surface is more activated towards resin adhesion [3].

Currently, the surface treatment used for zirconia restorations is by sandblasting, and in particular, also called by "tribochemical silica-coating". The latter employs silica-coated alumina sand particles to promote adhesion between resin cement and zirconia restorative materials [4]. A dental sandblasting unit used in dental laboratories is connected to pressurized air supply. The device directs a stream of silica coated alumina sand particles under compressed air onto the substrate surfaces. The impact of the sand particles on the surface causes them to embed onto the substrate surface. A thin silica coating is formed. This is followed by silanization by applying a silane coupling agent onto the surface and thereafter cementation with resin cement. A silane coupling agent contains two different functional groups that can connect the resin monomers and inorganic substrates [5]. However, this surface treatment method of zirconia induces subsurface damage which affects the mechanical properties of dental zirconia [6].

Different surface treatment methods have been reported in an attempt to improve the resin zirconia bonding without causing zirconia surface damage. Methods such as selective infiltration etching, nano-coating of alumina, internal coating technique, chemical vapor deposition [5] and sol–gel coatings [7] have been reported. These methods may alter the surface topography and (or) surface chemistry of zirconia which in turn affects the bonding of resin to zirconia to a different extent.

In the current study, another approach to surface treatment of zirconia to enhance resin zirconia bonding is reported. A silica coating is formed on zirconia surface by silicon nitride hydrolysis under alkaline condition at different immersion times. The hydrolysis of silicon nitride produces colloidal silica. The silica particles formed deposit on the zirconia surface. The coating is followed by a thermal treatment at 1400 °C. The coating was characterized by SEM, EDX, XRD and Raman. The shear bond strength of resin to zirconia using this surface treatment

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http://dx.doi.org/10.1016/j.msec.2014.10.029
0928-4931/

technique was evaluated under three storage conditions. The null hypothesis was that there is no difference in resin bond strength by different surface treatment methods and under different storage conditions.

2. Materials and methods

Unsintered zirconia blocks (Upcera, Liaoning, China) were cut into zirconia discs with dimension of 20 mm × 8 mm × 5 mm. The zirconia discs were sintered at 1480 °C for 3 h according to the manufacturer's instruction. The sintered zirconia samples were rinsed in 70% ethanol solution in an ultra-sonic bath for 10 min. After drying, the zirconia sample surfaces were polished with 220, 320, 500 and 1000-grit silicon carbide papers under running water using a polishing machine (Lunn Major, Struers, Denmark). The zirconia samples were cleansed ultrasonically for 10 min in 70% ethanol and rinsed with 70% ethanol. They were next air-dried at room temperature for 30 min.

2.1. Sandblasting of zirconia surfaces

A control group of zirconia samples were sandblasted using Rocatec Sand Plus (110 μm of silica-coated alumina particles, 3M ESPE). A constant pressure of 300 kPa for 15 s was applied at a perpendicular distance of 10 mm [8]. The samples were rinsed ultra-sonically in 70% ethanol for 10 min to remove any loosely embedded silica particles. They were dried in air for 30 min.

2.2. Surface treatment of zirconia samples by silicon nitride hydrolysis

The zirconia blocks were immersed into a 4 M sodium hydroxide (Sigma Aldrich) solution in a Teflon beaker. The solution was then heated up to 90 °C under continuous stirring. A 0.6 wt.% silicon nitride (nanopowder, 99%, US Research Nanomaterials, Houston, USA) dispersion was prepared by adding the nanopowder into the solution. The zirconia samples were immersed into the dispersion for 6, 24, and 48 h

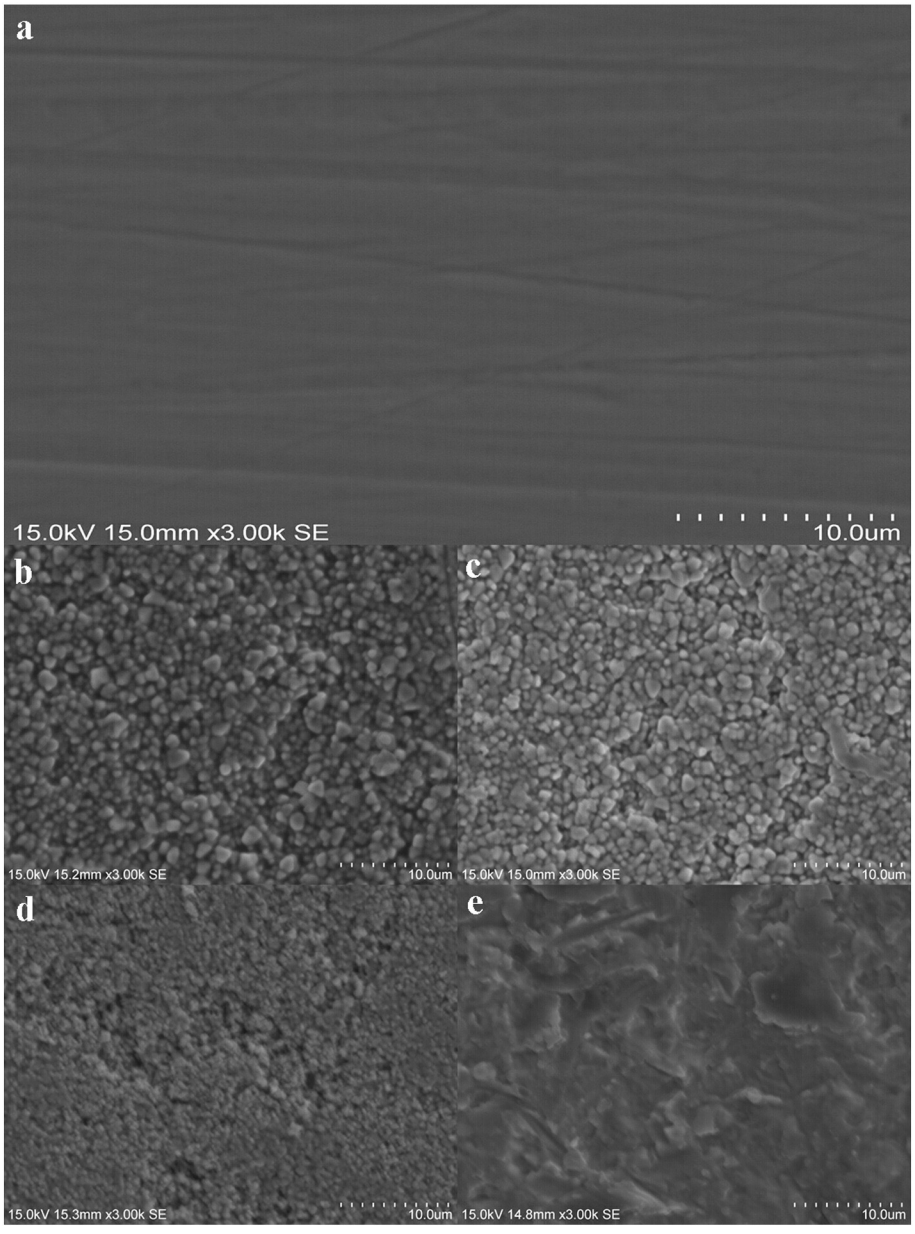

Fig. 1. SEM micrographs (3000×) of zirconia surfaces with various surface treatments: a. polishing, b. silicon nitride hydrolysis (6 h), c. silicon nitride hydrolysis (24 h), d. silicon nitride hydrolysis (48 h), and e. sandblasting.