Available online at www.sciencedirect.com

SciVerse ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/jden

Surface characterization of dental Y-TZP ceramic after air abrasion treatment

Lubica Hallmann [a,*], *Peter Ulmer* [b], *Eric Reusser* [b], *Christoph H.F. Hämmerle* [a]

[a] *Clinic of Fixed and Removable Prosthodontics and Dental Material Science, Center of Dental Medicine, University of Zürich, Switzerland*
[b] *Institute of Geochemistry and Petrology, ETH Zürich, Switzerland*

ARTICLE INFO

Article history:
Received 23 February 2012
Received in revised form
3 May 2012
Accepted 4 May 2012

Keywords:
Airborne particle abrasion
Dental zirconia
FESEM
XPS

ABSTRACT

Objective: The aim of this study was to characterize the surface of Y-TZP after abrasion with various airborne particles.
Methods: The Y-TZP blanks were cut into 44 discs and sintered according to the manufacturer's instructions. The specimens were treated as follows: (a) control specimens, (b) abraded with 50 μm alumina, (c) abraded with 110 μm alumina, (d) abraded with 30 μm silica-coated alumina, (e) abraded with 110 μm silica-coated alumina, (f) abraded with 110 μm alumina followed by 110 μm silica-coated alumina particles. Airborne abrasion was performed at a pressure of 2.5 bar for 15 s/cm^2. The Y-TZP was characterized using X-ray photoelectron spectroscopy (XPS), field emission scanning electron microscopy (FESEM) and X-ray diffraction analysis (XRD).
Results: Surface morphology of Y-TZP ceramic was changed after the airborne abrasion process compared to the control specimens. The grain boundaries disappeared and part of the airborne particles are embedded and/or rested on the ceramic surfaces. The elemental composition of the Y-TZP surface after the airborne abrasion process depended on the type and size of these particles. The concentration of Si resulted higher after the airborne abrasion process with 110 μm alumina followed by 110 μm silica-coated alumina particles in comparison to the specimens abraded with 110 μm silica-coated alumina particles. The ratio of elements normalized by yttrium for these specimens was: [Zr]/[Y]/[Al]/[Si] = 15.2/1.0/26.0/73.6, respectively.
Conclusion: The change of grain topography occurred during each impact process. Silica nano-particles covered not only loosely the abraded ceramic surface after abrasion process, but the release of kinetic energy in form of thermal energy resulted in melting of the ceramic surface and in the formation of zirconium silicate.

1. Introduction

During the last few decades, dental research concentrated on metal-free prosthetic restorations in order to improve the aesthetic outcome of ceramic fixed partial dentures (FPDs).[1] Among other ceramic materials, zirconia-based ceramics are expected to perform better in dental applications due to their high biocompatibility and superior mechanical properties as opposed to glassy matrix and alumina ceramics.[2,3] Yttria

* *Corresponding author at*: Clinic of Fixed and Removable Prosthodontics and Dental Material Science, Center of Dental Medicine, University of Zürich, Plattenstrasse 11, 8032 Zurich, Switzerland. Tel.: +41 44 632 3371; fax: +41 44 634 43 05.
E-mail addresses: lubica.hallmann@zzm.uzh.ch (L. Hallmann), peter.ulmer@erdw.ethz.ch (P. Ulmer), eric.reusser@erdw.ethz.ch (E. Reusser), christoph.hammerle@zzm.uzh.ch (Christoph H.F. Hämmerle).
0300-5712/$ – see front matter
http://dx.doi.org/10.1016/j.jdent.2012.05.003

stabilised polycrystalline tetragonal zirconia (Y-TZP) ceramic has the highest flexural strength (700–1200 MPa) and fracture toughness (7–10 MPa $m^{1/2}$) of all presently available dental ceramics.[4–8] The Y-TZP ceramics are indicated for full coverage single or multiple units as well as surface retained resin-bonded FPDs, where in the latter adhesion is of particular importance for the durability of such restorations.

A strong, stable resin bond to the ceramic surfaces is desirable for a long-term service of FPDs. This can be achieved by either micromechanical interlocking of the resin cement, and/or chemical bonding between the ceramic surface and the luting agent.[6,7,9–13] Surface conditioning with hydrofluoric or phosphoric acid etching or application of ammonium bifluoride that increase the micromechanical retention of resin based materials to silica-based ceramics, are not effective on the Y-TZP ceramics. Therefore, other conditioning methods such as airborne abrasion are suggested to achieve a strong and durable bonding of resin based cements to this ceramic.[9–13]

In principle, air abrasion cleans the surface, removes impurities, increases surface roughness, modifies the surface energy and wettability, and at the same time provides mechanical impinging of the particles on the surface. In that respect, the shape, size, morphology and chemical composition of the particles may play a very important role on the modification of the surface.[14–21]

Y-TZP consists of predominantly tetragonal phase (t), retained at room temperature by careful control of grain size and stabilizer concentration.[1–9] The transformation of the tetragonal to the monoclinic phase takes place under stress focusing at crack tips under applied stress, which is accompanied by a volume expansion of about 3–5%. This stress-induced phase transformation represents the toughening mechanism of Y-TZP, which increases its crack propagation resistance.[18–25] The tetragonal (t) – monoclinic (m) phase transformation is a diffusionless shear process at near sonic velocities, similar to those of martensitic texture in quenched steel.[19,25]

The most commonly used airborne particles in dental applications are alumina or silica-coated alumina particles.[9–13,18–23,26–38] When the abrasive particles hit the substrate surface, complex reactions may take place on the surface.[14–17] While some of the silica particles may be expected to react chemically with the ceramic surface others may loosely adhere on it. The concentration of elements present on the abraded surface may vary as a function of pressure and particle type which may, in turn, affect the adhesion of the ceramic-cement assembly.[31]

Air abrasion processes could either be performed in the laboratory or at chairside using air abrasion devices. It is currently not known to what extent the particle size affects the chemical composition of ceramic surface at a nano scale. Therefore, the objective of this study was to evaluate the chemical composition of the Y-TZP ceramic surface, and how the airborne particles were embedded on its surface after the impact process. The working hypothesis was that the elemental composition of Y-TZP ceramic surface is changed after air abrasion and the silica nano-particles employed for the air abrasion process do not only loosely adhere on the ceramic surface, but part of them react with the zirconia surface, which leads to a chemical bonding between silica nano-particles and ceramic surface (tribochemical effect).

2. Materials and methods

2.1. Specimen preparation

Y-TZP ceramic blanks (Cercon base, Cercon DeguDent, Hanau, Germany) (ZrO_2, 5% Y_2O_3, <2% HfO_2, <1% $Al_2O_3 + SiO_2$) were cut with a cutting machine (Precision Diamond Wire Saw, Well, Switzerland) into discs ($n = 44$, diameter: 25 mm, thickness: 3 mm) under copious water. They were subsequently sintered in a programmed furnace (Cercon heat, DeguDent, Hanau, Germany). The sinter temperature was set to 1350 °C for 2 h. The specimens were randomly divided into three main groups to be analyzed by X-ray Photoelectron Spectroscopy (XPS), Field Emission Scanning Electron Microscopy (FESEM) and X-ray diffraction (XRD).

The specimens for FESEM analysis were polished up to 1 μm with diamond suspensions (Struers, Ballerup, Denmark) and thermally etched while the specimens for XPS measurements were not polished, to avoid contamination of specimens by the polishing process. Air abrasion of ceramic surface was performed with 50 and 110 μm alumina particles (Al_2O_3, >99%) (Benzer Dental, Zürich, Switzerland, Rocatec® Pre, 3M ESPE, Seefeld, Germany), respectively. A commercial silica coating system was used to coat tribochemically the surface of Y-TZP ceramic with silica particles (CoJet™Sand, Rocatec® Plus, 3M ESPE, Seefeld, Germany) (A_2O_3, <3% amorphous SiO_2). Air abrasion was performed at a pressure of 2.5 bar for 15 s/cm^2 from a distance of 10 mm (CEMAT NT4, Wassermann, Hamburg, Germany).

After air abrasion, all specimens were first air-blown at a pressure of 2.5 bar for 1 min and then ultrasonically cleaned in isopropanol 99.8% (Merck, Darmstadt, Germany) for 20 min (Bransonic Ultrasonic Cleaner, Danbury, USA) at a frequency of 42 kHz.

2.2. Surface elemental composition

The surface chemistry of the specimens was investigated by X-ray photoelectron spectroscopy (XPS). The analyses were performed with a PHI Quantera SXM spectrometer (ULVAC-PHI, Chanhassen, MN, USA). All XPS spectra were collected at an emission angle of 45° using a beam size of 200 μm at a power of 50 W in the constant-analyzer-energy (CAE) mode. High resolution spectra were acquired using a pass energy of 55 eV and a step size of 0.1 eV (full-width-at-half-maximum (FWHM) of the peak height for Ag $3d_{5/2}$ 0.64 eV). Survey spectra were collected with a pass energy of 280 eV and a step size of 1 eV. A high-performance, floating-column ion gun and an electron neutralizer were used for charge compensation. The residual pressure in the analysis chamber was below 5×10^{-7} Pa. The spectrometer was calibrated according to ISO 15472:2001 with an accuracy of ±0.1 eV.

2.3. Elemental composition

Elemental compositions of non-abraded and abraded specimens ($n = 3$ for each group) were further investigated by EDS (energy dispersive X-ray spectroscopy). The data were acquired using a Carl Zeiss Supra 50 VP FESEM (Oberkochen,