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Interaction of zirconia primers with yttria-stabilized zirconia surfaces

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ABSTRACT

Objectives. Recently several zirconia primers have been introduced to mediate bonding of dimethacrylate resins to yttria-stabilized zirconia frameworks (Y-TZP). The aim of the study was to evaluate the changes in surface chemistry of Y-TZP frameworks induced by zirconia primer treatments.

Methods. Polished Y-TZP discs (Lava, 3M ESPE), ultrasonicated for 10 min in ethanol, water-rinsed and air-dried were treated as follows: A: Reference (no treatment), B: Treatment with Z Prime Plus (Bisco), and C: Treatment with Z-Bond (Danville Materials). The primer films formed on Y-TZP surfaces were air-dried, left intact for 5 days (dark storage, 37 °C, 40% RH), rinsed with 10 ml acetone to remove the loosely bound fractions, air-dried and studied by: (a) reflection optical microscopy, (b) reflection Fourier transform infrared microscopy (RFTIRM) and (c) scanning electron microscopy/energy dispersive X-ray microanalysis (SEM/EDX).

Results. An amorphous thick film was observed on primed and acetone rinsed Y-TZP surfaces after B treatment, whereas C treatment formed a thinner film with phase-separated aggregates. The RFTIRM study showed that both primers induced carboxylate salt formation on Y-TZP. Phosphate groups in a dissociative form have been identified on Y-TZP, as well, indicating formation of phosphate salts. EDX analysis showed increased C, O and P content on the films, which masked the substrate contributions.

Significance. The primers tested formed carboxylate and phosphate salts on Y-TZP, promoting thus chemical adhesion. However, the differences in the film forming properties and water solubility between the carboxylate and phosphate salts may affect the strength and the durability of adhesive resin interfaces with Y-TZP, as mediated by these primers.

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1. Introduction

Among the various emerging techniques for bonding dimethacrylate resins to yttria-stabilized zirconia (Y-TZP)

frameworks, zirconia primers have been introduced, integrating the current knowledge in dentin bonding and the advances in bonding to precious and base metal alloys [1,2]. The majority of these agents are based on phosphate compounds, since it has been established that stable Zr–O–P bonds can be

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formed between zirconia and phosphate or phosphonate solutions, providing a means of grafting phosphate species onto zirconia [3]. Acrylate or methacrylate functionalized phosphate monomers with a hydrophobic backbone have been considered as the most suitable coupling agents for zirconia surfaces based on the bond strength data obtained from various studies [1,2]. It has been reported that combined phosphate/dithione and phosphate/carboxyl monomers produce a synergistic effect on resin bonding to zirconia [1]. Phosphate-based zirconia primers are usually applied on mechanically roughened or alumina sandblasted surfaces [1,4], since for silica-rich tribochemically coated zirconia surfaces, silane coupling agents are indicated [1,5,6]. Nevertheless, in several modern silane primers, phosphate monomers have replaced the nonreactive acidic media (i.e. acetic acid) required for the acid-catalyzed hydrolysis of silanes, creating thus universal primers for glass and polycrystalline ceramics [6].

Despite the numerous bond-strength studies performed under a variety of testing and aging conditions, limited evidence exists for documentation of a chemical reaction between zirconia primers and zirconia surfaces. There is only a single paper reporting mass-spectrometry fragments corresponding to Zr–O–P formation on zirconia surfaces primed with a zirconia coupling agent containing phosphate and carboxylic monomers [7].

The aim of the present study was to investigate the bonding mechanism of two commercially available zirconia primers with 3% yttria-stabilized zirconia (Y-TZP) disk surfaces. The null hypothesis was that both primers cannot interact with the substrate by means of chemical bonding.

2. Materials and methods

The materials used in the study are listed in Table 1. The Y-TZP disks were metallographically polished in a grinding/polishing machine (DAP-V, Struers, Ballerup, Denmark) using 20, 15 and 6 μm (3000 grit size) diamond disks under water coolant and ultrasonicated for 5 min in ethanol to remove surface contaminants. The surfaces produced ($S_a = 41\text{--}70\text{ nm}$ and $S_z = 350\text{--}420\text{ nm}$ in min/max values, as measured with an optical profiler) were subsequently rinsed with distilled water, air-dried for 10 s and randomly classified in two groups of three specimens each. Half of the polished surface areas of the

specimens of each group were treated with the corresponding primer, according to the manufacturers' instructions, whereas the rest were used as untreated reference. All specimens were stored in dark conditions at 37 °C and 40% relative humidity for 5 days. Then, the surfaces were rinsed with 10 ml of acetone to remove the loosely bound fraction of the adsorbed components, air-dried as before and transferred for analysis.

The primed surfaces and their individual controls were observed under an optical microscope operated in bright-field reflection mode (DM4000B, Leica, Wetzlar, Germany) at 200X magnification. Then, they were examined by reflection Fourier-transform infrared microscopy (RFTIRM) employing an FTIR microscope (AutoImage, Perkin-Elmer, Bacon, UK) equipped with a mid-band mercury-cadmium telluride (MCT) detector, attached to an FTIR spectrometer (Spectrum GX, Perkin-Elmer). The optical system of the FTIR microscope, operated in bright-field reflection mode, was used to locate the regions with the adsorbed primer from where spectra were acquired under the following conditions: 300 $\mu\text{m} \times 300\text{ }\mu\text{m}$ aperture, 300 scans co-addition per spectrum, 4000–650 cm^{-1} wavenumber range, 4 cm^{-1} resolution and $\sim 1\text{ }\mu\text{m}$ sampling depth. Spectra acquisition and interpretation were performed by AutoImage 5.0/Spectrum 5.0.1 software (Perkin-Elmer). On each specimen two regions were analyzed to verify reproducibility. All the spectra were subjected to Kramers–Kroning and baseline corrections. Transmission spectra of 20 s air-dried primer films applied on Ge windows were used as reference. The 2000–850 cm^{-1} wavenumber range was defined as the region of interest, and the absorbance spectra recorded, were further subjected to 2nd derivative peak fitting for the ester ($\nu_{\text{C}=\text{O}}$, 1800–1650 cm^{-1}), carboxylate (asymmetric ν_{COO^-} at 1600–1500 cm^{-1} , symmetric ν_{COO^-} at 1450–1350 cm^{-1}) and phosphate (1200–950 cm^{-1}) peak domains. Peak fitting was performed after 5% smoothening (FFT filtering), employing Pearsons VII algorithm at standard width/variable shape mode and 2% zero baseline. Peak fitting analysis was performed by PeakFit v4.12 software (Seasolve, Framingham, MA, USA).

The same specimens were further subjected to high vacuum scanning electron microscopy/energy dispersive X-ray microanalysis (HV-SEM/EDX). The primed and untreated Y-TZP surfaces were sputter-coated with C in a sputter-coater unit (SDD 004 with OCD 30 attachment, Bal-Tec, Vaduz, Liechtenstein) and examined in a SEM (Quanta 200, FEI, Hillsboro,

Table 1 – The materials used in the study (composition according to manufacturers' information).

Product (batch)	Composition	Manufacturer
Zirconia primers		
Bisco Z-Prime Plus (1000012705)	Biphenyl dimethacrylate (BPDMA), 2-Hydroxyethyl methacrylate (HEMA), 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Aromatic substituted carboxylic acid, Ethyl alcohol	Bisco, Inc., Schaumburg, IL, USA
Danville Z-Bond (082-115A-5)	Pyromellitic dimethacrylate (PMDM), 2-Hydroxyethyl methacrylate (HEMA), Bisphenol-A glycidyl methacrylate (BisGMA), 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Glycerol dimethacrylate-succinate (GDMAS), Ethyl alcohol	Danville Materials, Inc., S. Ramon, CA, USA
Zirconia specimens		
Lava	3% mol yttria stabilized tetragonal zirconia (3Y-TZP)	3M ESPE, Seefeld, Germany

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