Chemical Analysis of Zirconia Surfaces after Tribochemical Silica Coating and Various Adhesives Treatments by ToF-SIMS

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I. Objective:

Zirconia is a polycrystalline ceramic free from glass, exhibiting chemical resistance to etching and silane treatments. To improve bonding of zirconia restorations, airborne-particle abrasion (AA) following by MDP or other chemical agents are the common methods. These methods aim to promote the formation of Zr-O-P and Si-O-Si bonds between zirconia and resin cements. The tribochemical silica coatings (TSC) may create micromechanically roughened surface and chemical modification, and its bonding stability is recommended. However, there exists a debate whether MDP- or silane-based chemical agent is suitable for TSC treated zirconia. The objective of this study was to perform a chemical analysis of the zirconia surfaces after TSC and various adhesives treatments by time-of-flight secondary ion mass spectrometry (ToF-SIMS).

II. Materials & Methods:

Zirconia disks were divided into 2 groups to receive different blasting treatments: AA, airborne-particle abrasion with 50 μm alumina particles for 15 s; and TSC, Rocatec soft (3M EPSE) treatment for 15 s. After blasting treatments, each disk was treated with one of three experimental primers (E-M, 5% experimental MDP solution, E-S, 5% experimental silane solution, and E-MS, the mixture of MDP and silane solution), or three commercial primers (SE, SE Bond Primer (Kuraray); RCP, RelyX Ceramic Primer (3M ESPE), and CCP, Clearfil Ceramic Primer Plus (Kuraray)). Three commercial primers represent products containing 10-MDP, silane, and a combination of 10-MDP and silane, respectively. Subsequently, these specimens were examined under ToF-SIMS (PHI TRIFT IV, ULVAC-PHI). The formation of Zr-O-P bond was examined by the related ions, and the silane-related ions were also examined.

III. Results:

The zirconia disks treated with the combined TSC and MDP-based primers (E-M and SE) showed greater Zr-P-O containing ions compared to those treated with combined AA and MDP-based primers. The ratio of Zr-P-O containing ions decreased upon the addition of silane, and the affected conditions were the most significant in group TSC/CCP. The siloxane comprising $Si_2O_5H_3^+$, $Si_3O_7H_3^+$, and SiOSi were calculated for Si-O-Si bonds. For TSC-treated disks, both experimental and commercial silane-based primers exhibited higher proportions of siloxane/Si-related ions (21% in E-S and 25% in RCP) compared to the MDP-silane combination groups (14% in E-MS and 16% in CCP). However, they were all higher than those detected in the AA/E-MS (7%) and AA/CCP (10%). The formation of siloxane was originated from the interaction of silane and TSC coating since only a few (1%) siloxane bonds were detected in the disk receiving TSC but without any silane-based primer application.

IV. Conclusion:

TSC treatment does not hamper the formation of Zr-O-P bonding, but also enhance the bond formation compared to AA. The TSC coating also react with silane to provide siloxane bonding. The Zr-O-P bonding established by MDP-based primer would be interfered by the coexistence of silane. The formation of siloxane bond was also impaired by MDP.

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Surface Characterization of 4Y-Zirconia after Hydrofluoric Acid Treatment

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I. Object: To investigate the surface morphology, elemental composition, and etching rate of 4Y-zirconia after hydrofluoric acid (HF) treatment.

II. Materials & Methods: 4Y zirconia powder (Zpex 4 White) was purchased from Tosoh Corporation. The powder was pressed with 200 kgf/cm^2 to form a disk and then cut to 14×14×4 mm in size. Subsequently, the specimens were kept at 1500 ^{o}C for 2h with a heating rate of 600 ^{o}C/hour to achieve 6 g/mm^3 density. The specimens were treated using 25% HF for 30, 60, and 120 mins at room temperature. Scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and Contact Surface Profilometer were used for surface characterization.

III. Results: SEM examination revealed that the etching depth increased with the increase of etching time. The mean etching depth was 0.7 μm after 60 mins treatment. The XPS scan of the full spectrum demonstrated the presence of hafnium (Hf) in addition to the presence of zirconium (Zr) and yttrium (Y).

IV. Conclusion: It is well known that HF is not effective for zirconia etching. The etching effect demonstrated in this study was possibly from the dissolution of hafnium dioxide by HF.

Surface Modification on Titanium Using Micro-arc Oxidation and Hydrothermal Treatment

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I. Object

Dental implants are widely used in the treatment of missing teeth nowadays. Implant surfaces are important for achieving better osteointegration and long-term survival rates. Micro-arc oxidation is an electrochemical surface modification that produce a porous oxide film by using high voltages. Hydrothermal treatment is a way to synthesis crystals at high temperature and pressure

II. Materials and Methods

The micro arc oxidation and hydrothermal treatment were used to modify the implant surface of pure titanium (Ti). The titanium discs were divided into 3 different surface roughness (Ra 0.5μm, Ra 1.2μm and Ra 3μm). The anodic oxide film with calcium and phosphate was formed using micro-arc oxidation with different voltage (300V and 400V), and hydroxyapatite crystals were created after 12 hours and hydrothermal treatment.

III. Results

The surface morphology was observed using scanning electron microscope (SEM) after micro-arc oxidation and hydrothermal treatment. Energy-dispersive X-ray spectroscopy (EDS) was used to analyzed the chemical elements of the materials. After micro-arc oxidation, the craters on anodic oxide film on titanium surface were smaller in 300V group than 400V group. With increased surface roughness, the density of craters was slightly decreased. After 12 hours of hydrothermal treatment, the hydroxyapatite crystals were formed, the amount of crystal was more in 400V group than 300V group. With increased surface roughness, the density of crystals was increased. According to the analysis of energy-dispersive X-ray spectroscopy (EDS), the anodic oxide film was composed mainly of Ti and O, with Ca and P in small percentages. After hydrothermal treatment, Ca, P, and O was increased.

IV. Conclusions

The micro-arc oxidation following 12 hours of hydrothermal treatment could form hydroxyapatite crystals on Ti surface.

Comparison of Polymerization Shrinkage Patterns of Bulk-fill and Conventional Composites by Digital Image Correlation Method

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Purpose:

Bulk-fill composites have been widely used in recent years. Compared with conventional composite resin, bulk-fill composites exhibit less polymerization shrinkage stress and better light transmission. The aim of this study was to investigate the time-dependent polymerization shrinkage patterns of bulk-fill and conventional composites of different viscosities using digital image correlation method and compare the shrinkage patterns between bulk-fill and conventional composites of both high and low viscosity.

Method:

Four resin composites from the same manufacturer were chosen to test: one low viscosity bulk-fill composite Filtek Bulk Fill Flowable (BFF), one high viscosity bulk-fill composite Filtek One Bulk Fill (OBF), one flowable composite Filtek Z350XT Flowable (Z350F), and one conventional nanocomposite Filtek Z350 (Z350). The composite material was filled into a slot (3 mm wide, 2 mm high, and 5 mm long) in a metal jig individually, with a coat of Vaseline to prevent adhesion. The specimen surface was sprayed with powders to produce sufficient contrast, allowing the tracking of individual points on the surface. The composites were light cured with irradiances of 1000mW/cm^2 (Smartlite Focus, Dentsply) through the lateral window of the slot for 40 s. A light microscope recorded the deformation of the composite specimen from the top before and after light curing at intervals of 5 s,10 s,15 s,20 s, 25 s,30 s, 35 s, 40 s. Subsequently, these images were input into a digital image correlation software (Vic-2D, Correlated Solutions Inc.) to analyze and calculate polymerization shrinkage strain and time-dependent changes.

Results:

In the analysis, it was found the Z350F exhibited the highest mean strain value after 40 s, followed by BFF, Z350 whereas OBF demonstrated the lowest value. OBF and Z350 showed steadily increased strain during curing, but BFF and Z350F showed abrupt increases at the end of curing. In BFF, the max. strain occurred at the bottom of composite and directed outward. In OBF, maximal strain occurred at the surface and directed downward. In Z350F, the max. strain moved from 1 to 2mm deep and directed outward. In Z350, the max. strain moved from 2 to 3mm deep and also outward.

Conclusions:

The analysis revealed that the high and low viscosity bulk-fill composites exhibited different shrinkage behaviors. Low-viscosity bulk-fill and flowable composites showed continuous composite shrinkage movements through curing, while high viscosity ones exhibit earlier gelation. Furthermore, the location of max. strain values varied among these composites. These findings suggest that high-viscosity bulk fill composite may offer advantages over conventional composite resins in terms of reduced polymerization shrinkage and less interfacial debonding

Biomechanical Analysis of Endocrowns with Different Margin Designs

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I. Objective:

The restoration of endodontically treated teeth is a crucial concern in dentistry. The progresses in ceramic materials and adhesive techniques have introduced the endocrowns restoration as a viable alternative. Nevertheless, there is an ongoing debate regarding the designs and preparations of endocrowns. Furthermore, the recent introduction of the "compression dome" concept reinforces the significance of preserving supporting structures to minimize the risk of tooth and restoration fractures. In this concept, restorations above the inflection line (usually the height of contour of the teeth) locate in a compression zone thereby requiring less retentive preparations. Hence, this study aimed to investigate the biomechanical behaviors of teeth receiving endocrowns with different margin designs in the context of the "compression dome" concept.

II. Material & Method:

32 intact human molars of similar sizes were collected for the study. These teeth were first endodontically treated and filled with GP points to leave 6mm coronal pulp space. The root of each tooth was coated with a thin layer of light body impression materials to simulate PDL, and then the teeth were planted vertically in epoxy resin. These teeth were divided into four groups (n = 8) to receive endocrown preparations with different margin locations and designs. For groups I and II, the margins of endocrowns were 2 mm above the inflection line but the margin was either butt-joint or with 1-mm ferrule around. For groups III and IV, the margins were 2 mm below the inflection line and the margin was either butt-joint or with 1-mm ferrule around. The outer axial walls were 12° taper, and the inner wall was taper. After crown preparation, the teeth received scanning then corresponding IPS e.max CAD endocrowns were fabricated. All endocrowns shared the same occlusal anatomy, and were cemented to teeth using their corresponding resin cements. After storage for 24 h, the samples were fixed at a 15° inclination on a universal testing machine. A static loading of 100 N was applied to the buccal cusps using a 6 mm diameter stainless steel spherical piston. During loading, a camera captured the images for the digital image correlation (DIC) analysis to assess the strain patterns of the restorations and teeth. Additionally, these teeth received cyclic loading of 100 N. The replicas of these crowned teeth were examined using SEM for their marginal integrity.

III. Results:

The DIC analysis showed that there were greater strain patterns in group I followed by group II, III, and IV. Before loading, two butt-joint margin groups had better marginal fit. After loading, the marginal gaps were greater in lingual margins compared to those in buccal margins. Groups I and II showed greater gaps compared to groups III and IV.

IV. Conclusion:

The results did not conform with the preposition of "compression dome" concept since the supra-inflection line groups showed greater deflection and less marginal integrity.

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Influences of Cavity Design on Biomechanical Behaviors of Zirconia and Lithium Disilicate Overlays

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I. Objective:

Bonded porcelain restoration is gaining popular in dental clinics. Lithium disilicate (LS2) has been chosen for fabricating overlays for its translucency and excellent bond strength through adhesive treatments. However, LS2 overlays showed higher fracture prevalence. Zirconia (Zr) is considered as the alternative for overlay fabrication. For two ceramics overlays, there is no evidence that certain cavity design has the best performance. Hence, this study aimed to investigate the effect of cavity design on the biomechanical behaviors of LS2 and zirconia overlays using a finite element analysis.

II. Material & Method:

An intact human molar was selected and scanned under a micro-CT (Bruker Skyscan 1276). The data was imported into the Mimics software (Materalise) to delineate the contours of dentin, enamel, and pulp, and then transformed into solid formats in a Geomagic software. At the meantime, resin teeth were printed out by a 3D printer to undergo four overlay preparations as: occlusal reduction only (O), occlusal reduction and boxes on two proximal surfaces (OB), OB preparation plus shoulder margins (OSB), and occlusal reduction, a central isthmus, and shoulder margins (OSI). The prepared teeth were scanned by an intraoral scanner to generate solid formats, and then combined with the sound tooth model to form four models of different overlays and their cement layers. These models were imported into a finite element analysis (ANSYS 2022). The overlay materials were assigned as either Zr or LS2, and then eight models were meshed using 0.2 mm elements. 600 N vertical force was applied on occlusal surfaces through a 6 mm diameter ball. The maximum von-Mises (MVM) stress and shear stress in the overlays, cements, and tooth were solved in eight models.

III. Results:

The MVM stress in groups O, OB, OSB, and OSI occurred at the force loading areas. The MVM values in four Zr overlays were higher than their corresponding LS2 groups except OSB. For each ceramic, O and OB had higher MVM stress, while OSI and OSB had lowest MVM stress. The MVM stress in all models were lower than the flexural strengths of their corresponding ceramics (Zr and LS2).

In eight groups, the shear stress of cements was present at the angle of the preparation design. Group O showed the lowest shear stress among the groups, while group OB and OSB showed the highest max. shear stress.

IV. Conclusion:

With the present results, the preparation design with boxes or isthmus will cause VM stress concentration, but shoulder margin design may reduce stress. The box or isthmus design may also increase the shear stress of cement. Although stress concentration appears in two ceramics, the values are under the strength limit of two ceramics.

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Evaluations of Tissue-Dentin Adhesives Consisting of Isobutyl Cyanoacrylate and Octyl Cyanoacrylate

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I. Objective:

Variety of tissue adhesives have been developed in recent years. The cyanoacrylate adhesives are considered most suitable for application in oral cavity to promote tissue adhesion or wound healing. Depending on the side chain length, cyanoacrylate adhesives exhibit different properties. Those with short chains can form tight and stronger bonds rapidly, while those with long chains have better biocompatibility. For root coverage surgery, a dentin-tissue adhesive may benefit the fixation of gingiva tissue. Hence, this study was aimed to evaluate the bond strengths of tissue adhesives consisting of isobutyl cyanoacrylate and octyl cyanoacrylate adhesives of different proportions to dentin surface using an *in vitro* test.

II. Material & Method:

Short-chain (4 carbons) isobutyl cyanoacrylates and long-chain (8 carbons) octyl cyanoacrylate (both from StarSpeed, China) were blended in different proportions to create five adhesives as: ❶ 100% isobutyl cyanoacrylates, BC; ❷-❹ mixture of isobutyl cyanoacrylates and octyl cyanoacrylate at different ratios (70%:30%, 50%:50%, and 30%:70%, respectively) as BC70OC30, BC50OC50, and BC30OC70; and ❺ 100% octyl cyanoacrylates, OC. A commercial tissue adhesive PeriAcryl®90HV (PA) (GluStich Inc., Canada) was used as a comparison. Human tooth roots were embedded and ground flat. The bond area was set as a circle of 4 mm diameter (12.56 mm^2). 15 μL of an adhesive was applied on the tooth roots, and then bovine gingiva was attached and compressed by 500 g force for 5 min. Lap shear bond strength was measured by pulling the gingiva from roots by employing an Autograph Universal Testing Machines (Shimadzu, Japan). The failure patterns were observed under an optical microscope after bond strength testing.

III. Results:

The results suggest a positive correlation between the ratios of isobutyl cyanoacrylate and bond strength. BC showed the best performance, and OC presented the lower bond strength among five experimental adhesives. The commercial tissue adhesive PA had inferior bond strength compared to any of five adhesives. The failure patterns analysis revealed that adhesive failure was the predominant failure mode for all type of adhesives.

IV. Conclusion:

With the present results, the ratio of isobutyl cyanoacrylate adhesive affects the immediate bond strength. The result suggests that short-chain isobutyl cyanoacrylate adhesive has stronger bonding to root dentin than long-chain octyl cyanoacrylate adhesive. However, the late (after 24 h) bond strength and the degradation of the adhesives still require evaluations in further research.