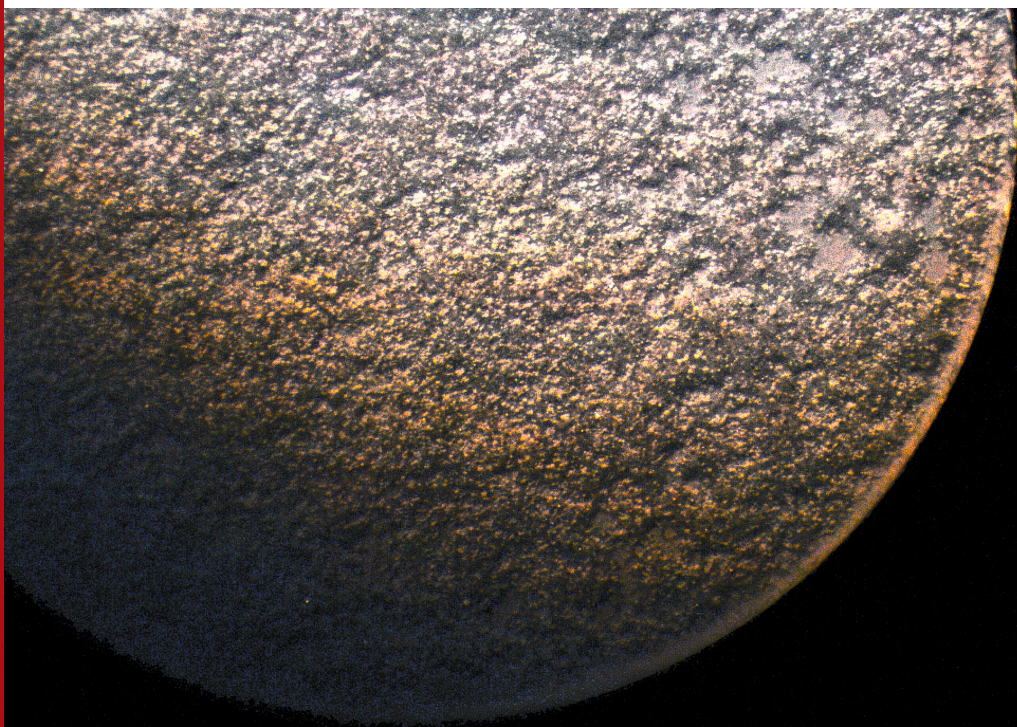


EVAGGELIA PAPIA

MICROMECHANICAL RETENTION AND CHEMICAL BONDING TO POLYCRYSTALLINE DENTAL CERAMICS

Studies on aluminum oxide and stabilized zirconium dioxide



MALMÖ UNIVERSITY

**MICROMECHANICAL RETENTION AND CHEMICAL BONDING
TO POLYCRYSTALLINE DENTAL CERAMICS**

Malmö University
Faculty of Odontology Doctoral Dissertations 2014

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Photographs and illustrations: Evaggelia Papia
ISBN 978-91-7104-539-3 (print)
ISBN 978-91-7104-540-9 (pdf)
Holmbergs i Malmö, 2014

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dioxide

Malmö University, 2014
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Malmö, Sweden

This publication is also available in electronic format at:
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To my family

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LIST OF PUBLICATIONS

This thesis is based on the following articles, referred to in the text by their Roman numerals. All articles are reprinted with permission from the copyright holders and appended to the end of the thesis.

- I. Papia E, Vult von Steyern P. Bond strength between different bonding systems and densely sintered alumina with sandblasted surfaces or as produced. *Swed Dent J* 2008; 32: 35–45.

Contribution of E. Papia: Planning the study, executing all tests, and writing the article (all steps with support).

- II. Papia E, Zethraeus J, Ransbäck P-Å, Wennerberg A, Vult von Steyern P. Impaction-modified densely sintered yttria-stabilized tetragonal zirconium dioxide (Y-TZP): Methodology, surface structure and bond strength. *J Biomed Mater Res Part B Appl Biomater* 2012; 100: 677-84.

Contribution of E. Papia: Planning the study; specimen preparation, bond strength testing and surface analysis (with support); and writing the article.

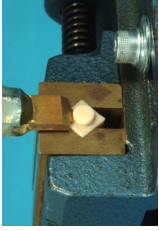
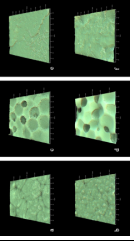
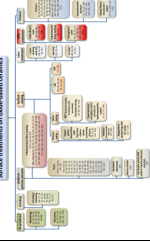

- III. Papia E, Larsson C, du Toit Madeleine, Vult von Steyern P. Bonding between oxide based ceramics and adhesive cement systems: A systematic review. *J Biomed Mater Res Part B Appl Biomater* 2014; 102: 395-413.

Contribution of E. Papia: Planning the study; literature search and data interpretation (with support); and writing the article.

IV. Papia E, Jimbo R, Chrcanovic BR, Andersson M, Vult von Steyern P. Surface structure and mechanical properties of impact-modified Y-TZP. Submitted

Contribution of E. Papia: planning the study, specimen preparation, biaxial flexure strength testing; surface analysis (with support); and writing the article.

Thesis at a glance

Study	Aim	Methods	Illustration	Main findings
I Bond strength between different bonding systems and densely sintered alumina with sandblasted surfaces or as produced.	Evaluate the shear bond strength between various adhesive cement systems and densely sintered alumina oxide, as-produced or sandblasted.	Shear bond strength test		No general recommendation can be made whether to use densely sintered alumina, either as-produced or surface treated with airborne particle abrasion, because bond strength depends on the adhesive cement system used.
II Impact-modified densely sintered yttria-stabilized tetragonal zirconium dioxide (Y-TZP): Methodology, surface structure and bond strength.	Describe a method for producing bondable Y-TZP structures using impact modification with two different mediums: glass granules and polymer granules. Investigate and describe the surface structures of surface-modified Y-TZP and evaluate the shear bond strength.	Shear bond strength test Surface analysis: Interferometry (IFM)		Impact modification with either glass granules or polymer granules can create a bondable cementation surface suitable for Y-TZP-based reconstructions, resulting in rougher surface structure compared to unmodified surfaces.
III Bonding between oxide-based ceramics and adhesive cement systems: A systematic review.	Make an inventory of existing methods for achieving bondable surface on oxide ceramics and evaluate which methods might provide sufficient bond strength.	Systematic literature review		There is no universal surface treatment for clinically sufficient bonding based on the oxide ceramics tested by the studies included in this review. Considerations should be given to the specific materials to be cemented and the adhesive cement systems to be used.
IV Surface structure and mechanical properties of impact-modified Y-TZP.	Investigate and describe the chemical surface composition of surface-modified Y-TZP and evaluate the flexural strength of Y-TZP, with or without surface modification, with various pretreatments: etching before or after sintering – alone and in combination with an adhesive cement system.	Flexural strength test Surface analysis: IFM, AFM, SEM, EDS, XRD		The surface structure and the chemical composition of glass-modified Y-TZP differ from unmodified Y-TZP. The flexural strength decreased with glass modification, but increased after cementation. The glass modification creates a bondable cementation.

ABSTRACT

Researchers are constantly developing new dental materials to replace missing teeth. One material group receiving major focus is ceramic materials; more specifically, oxide ceramics; and, in particular, yttrium dioxide-stabilized tetragonal polycrystalline zirconium dioxide (Y-TZP). In addition, one of the major challenges is to ensure retention of oxide ceramic-based restoration in the mouth, in a tissue preserving way.

Success in traditional cementation of dental restorations relies on a geometric form that establishes the macromechanical retention, the surface structure of the dental restoration, the tooth substance (micromechanical retention) and the cement itself. In clinical situations when macromechanical retention is insufficient, it may be necessary to use an adhesive cementation technique. Reliable adhesive bonding between the restoration, the cement, and the tooth substance requires micromechanical retention and cement that achieves chemical retention. In oxide ceramics, chemical retention has been difficult to achieve and unpredictable. Various techniques have been proposed for modifying the surface of oxide ceramic-based restorations making adhesive cementation technique a possible treatment option.

The overall aim of this thesis is to evaluate and develop techniques for modifying the surface of oxide ceramics that enable durable bonding between the restorations and adhesive cement systems. Additionally, the thesis will inventory existing methods for achieving a bondable surface on oxide ceramics and how these methods affect the materials.

Study I evaluated bond strength between several adhesive cement systems and densely sintered aluminum oxide. Two of six of the cement systems studied showed acceptable bonding to densely sintered aluminum oxide. The choice of surface treatment for the oxide ceramic should be based on the cement system to be used.

Study II described a modified-additive technique for producing bondable Y-TZP and evaluated the resulting surface structure and bond strength. Surface-modified Y-TZP showed a rougher surface structure and higher bond strength than unmodified Y-TZP. Study IV extended these evaluations with additional surface analysis and flexural strength testing. The results showed increased surface roughness, with a chemical composition of glass and with a content of monoclinic phase. Compared to unmodified Y-TZP, glass-modified Y-TZP showed lower flexural strength values that increased with the use of cement.

Study III was a systematic literature review to inventory existing methods for achieving a bondable surface on oxide ceramics. This study also evaluated which methods provide clinically relevant bond strength and classified the various surface treatments into seven main groups: as-produced, grinding/polishing, airborne particle abrasion, surface coating, laser treatment, acid treatment, and primer treatment. Abrasive surface treatment, as well as silica-coating treatment, combined with the use of a primer treatment can result in sufficient bond strength for the bonding of oxide ceramics. This conclusion, however, needs to be confirmed by clinical studies. There is no universal surface treatment; the choice should be based on the specific materials.

Together, the results of this thesis demonstrate that different surface treatments/modifications of oxide ceramics increase the bond strength between ceramics and adhesive cement systems. Surface modification with a glass medium was particularly effective. All surface treatment, however, affects the material properties and the resulting dental restoration. Choice of surface treatment should be made based on the restoration materials: the oxide ceramics and the adhesive cement systems.

POPULÄRVETENSKAPLIG SAMMANFATTNING

Det pågår en ständig utveckling av olika dentala material avsedda för att ersätta förlorad tandsubstans. En materialgrupp som är särskilt intressant är höghållfasta keramiska material, oxidkeramer så som yttriumdioxidstabiliserad tetragonal polykristallin zirkoniumdioxid (Y-TZP). Förutom utveckling av material, är en av de större utmaningarna att få tandersättningar att sitta fast i munnen på ett vävnadsbesparande sätt.

Vid traditionell cementeringsteknik, fästs tandstödda ersättningar med vattenbaserade cement, vars vidhäftning dels är beroende av att tänderna slipas i syfte att skapa en geometrisk form för att åstadkomma makromekanisk retention och dels av ytstrukturen på tand och tandersättning som skapas under processen och utgör mikromekanisk retention. I kliniska situationer med otillräcklig makromekanisk retention kan det vara nödvändigt att använda adhesiv cementeringsteknik. En förutsättning för en tillförlitlig adhesiv bindning mellan tandersättning, cement och befintlig tand är mikromekanisk retention och resinbaserade cement som möjliggör en kemisk bindning. Det senare har visat sig vara svårt och oförutsägbart att uppnå för oxidkeramer. Olika tekniker för modifiering av oxidkeramers cementeringsyta har föreslagits för att möjliggöra adhesiv cementeringsteknik.

Övergripande mål med föreliggande avhandlingsarbete var att utveckla och utvärdera metoder för att modifiera polykristallina keramers yta och därigenom möjliggöra kombinerad mekanisk och kemisk bindning mellan oxidkeramer och adhesiva cementsystem.

I delarbete I utvärderades bindningsstyrkan mellan olika adhesiva cementsystem och en tätsintrad aluminiumoxidbaserad keram. Två av sex undersökta cementsystem uppvisade acceptabel bindning till aluminiumoxid. Valet av ytbehandling på oxidkeramen bör baseras på vilket cementsystem som ska användas.

I delarbete II presenterades och utvärderades en ny framställningsteknik för ytmodifierad Y-TZP, lämpad för adhesiv cementeringsteknik. Ytmodifieringen visade ökad mikrostruktur och högre bindningsstyrka jämfört med obehandlad Y-TZP. Uppföljning gjordes i delarbete IV med ytterligare ytanalyser och hållfasthetstest. En kemisk sammansättning med glas och monoklin fas identifierades med ökad ytråhet. Ytmodifieringen med glasmedium resulterade i lägre hållfasthet som dock ökade i samband med cementering.

Delarbete III var en systematisk litteraturoversikt med syfte att inventera olika metoder för ytbehandling/modifiering av oxidkeramer och utvärdera vilka av dessa som ger kliniskt relevant bindningsstyrka. Indelningen av de olika ytbehandlingarna var: fabriksproducerad, slipad/polerad, sandblästrad, ytmodifierad med olika typer av täckande lager, laser-, syra- och primerbehandlad. Sandblästring eller kiseltäckning av cementeringsytan kombinerat med primer utmärkte sig med högre värden på bindningsstyrkan, något som dock ännu inte blivit bekräftat i kliniska studier. Det finns ingen universell ytbehandling. Valet av ytbehandlingar bör baseras på vilket material som ska användas.

Sammanfattningsvis visar resultaten i avhandlingen att olika ytbehandlingar av oxidkeramer, i synnerlighet ytmodifiering med glasmedium, kan öka bindningsstyrkan mellan keram och adhesivt cementsystem. All ytbehandling påverkar dock materialets egenskaper och slutligen tandersättningen. Valet av ytbehandling bör göras utifrån specifika materialval, avseende både keram och respektive cementsystem.

ABBREVIATIONS AND DEFINITIONS

AFM	Atomic force microscopy
Bis-GMA	bisphenol-A-diglycidyl-methacrylate
CAD	Computer-aided design
CAM	Computer-aided manufacturing
CIP	Cold isostatic pressing
EDS	Energy-dispersive X-ray spectroscopy
FDP	Fixed dental prosthesis
HF	Hydrofluoric acid
HIP	Hot isostatic pressing
IFM	Interferometry
LTD	Low temperature degradation
MDP	10-methacryloyloxydecyl-dihydrogen-phosphate
MPa	Megapascal
μ SBS	Micro-shear bond strength
μ TBS	Micro-tensile bond strength
N	Newton
PFM	Porcelain fused to metal
RBCB	Resin-bonded all-ceramic bridges
RDP	Removable dental prosthesis
RT	Room temperature
TBS	Tensile bond strength
TC	Thermocycling
TEC	Coefficient of thermal expansion
SBS	Shear bond strength
SEM	Scanning electron microscopy
XRD	X-ray diffraction

Y-TZP	Yttrium oxide-stabilized tetragonal zirconium dioxide polycrystals
Alumina	Aluminum oxide
Yttria	Yttrium oxide
Zirconia	Zirconium dioxide

INTRODUCTION

Loss of teeth lowers self-esteem and impairs oral function. Treating tooth loss is very important for those who are affected. Patients who receive treatment experience increased self-esteem and improved oral function (1) and quality of life (2). There are a variety of treatment options to replace missing teeth, with fixed dental prostheses (FDPs) or removable dental prostheses (RDPs). Many patients prefer FDPs, either tooth- or implant-supported (3).

Treatment planning

The increased demand for aesthetic and biocompatible materials, together with the development of high-strength ceramics and new technological process, has made the use of all-ceramic materials for FDP treatment a common choice for the patient and dentist (4, 5). But treatment decisions include more than choosing the dental material for the restoration. It also involves patient preference and the prevailing clinical conditions, which can affect the design of the tooth preparation and restoration, and subsequently the choice of cementation technique (6, 7). Tooth preparation depends on the quality and quantity of the tooth substance remaining, the space needed for the intended restorative material, and the expected load under function in the oral cavity (6). Both the prepared tooth and the restoration should have smooth and rounded contours to avoid concentrations of stress (8). Replacement of missing tooth tissue should restore function with minimal biological cost (i.e., tooth preserving treatment) while establishing retention and resistance, providing strength and internal and marginal fit between the supporting tooth substance and the restoration. Tooth preparation

has the risk of injuring the pulp and surrounding gingival tissues. Excessive tooth reduction may lead to endodontic complications from increased temperature and dehydration. Inadequate removal of tooth substance, however, may cause an over-contoured restoration, which could affect aesthetics and lead to biological complications because of difficulties maintaining oral hygiene (6).

Restorations are retained with either traditional non-adhesive water-based cements (often referred to as conventional cements) or resin-based adhesive polymerizing cements (9). The traditional cementation technique is based on the macromechanical retention gained from the geometry of the preparation, which provides retention and marginal fit, and influences the durability of the restoration (8,10). The most commonly used water-based cement is zinc phosphate, which is considered the criterion standard due to its successful long-time clinical use. Adhesive cementation techniques promote preservation of dental tissue because they rely on micromechanical and chemical retention, and not on macromechanical retention. Well-established bonding between the interfaces of the restoration and cement, and the cement and the tooth, improves the retention and marginal seal in comparison to cement that relies on macromechanical retention (7, 9, 11). It is preferable for the preparation site to be completely in enamel to achieve and maintain an optimal bond. Preparation, however, often exposes considerable amounts of dentine. The dentine bond is more complicated than the bond to enamel because of the characteristics of dentine, which include its lower inorganic content, its tubular structure, and variations in this structure. Therefore, bond strength to enamel is higher and more predictable than to dentine (12).

Choosing materials

Dental ceramics

Ceramics are used in dentistry because they closely mimic the optical properties of enamel and dentine, in addition to their chemical and mechanical properties such as biocompatibility, high elastic modulus, low thermal expansion coefficients, and good wear resistance (13). All these properties arise from the strong covalent and ionic interatomic bonds of ceramics. Unfortunately, some of their mechanical properties are undesirable, such as brittleness. If

they are deformed more than 0.1-0.3 %, they will fracture (14, 15). Another disadvantage is that ceramic materials are sensitive to pre-existing flaws and defects, both on the surfaces and within the material. Flaws and defects can act as starting points for crack formation and, under load, lead to crack propagation that affects the strength of the material (16, 17).

According to standards set by the International Organization for Standardization (ISO), ISO 6872:2008 Dentistry - Ceramic materials (18), dental ceramics have two classifications: ceramic products that are provided from powder (Type I), and all other forms of ceramic products (Type II). Other classification methods involve intended use, chemical composition, process technology (19) or sensitivity to hydrofluoric acids (HF) (20). This thesis classifies ceramics according to their chemical composition: porcelain, glass ceramics, hybrid ceramics, and oxide ceramics (Table 1) and their intended use.

Due to their high glass content, porcelain and glass ceramics are esthetic materials with desirable optical properties, but limited strength. These two dental ceramics can be used monolithic (full-contour) restorations or as (surface) porcelain/ceramic veneer material in metal- or oxide ceramic-based restorations. Monolithic restorations made of porcelain are used only for laminate veneers in the anterior region. Glass ceramics have a wider application. The indications for glass ceramics differ depending whether they are leucite-based or lithium disilicate-based. The latter shows higher strength (21). Leucite-based glass ceramics can be used for laminate veneers, crowns in the anterior region and as onlays and inlays in any regions of the mouth. Glass ceramics based on lithium disilicate have these same indications but can also be used as monolithic restorations for FDPs of up to three units in the anterior and premolar regions (4, 21). The material shows approximately half the strength compared to yttrium-stabilized tetragonal zirconium dioxide polycrystals (Y-TZP), but it has three times the strength of the veneering porcelain that is used in combination with Y-TZP (22, 23). Even if glass ceramics have greater strength and toughness compared to porcelain, they both need to be etched and adhesively cemented to reinforce the ceramic restoration to withstand loads during function (24, 25).

Table 1. Overview of ceramic materials. Classification, mechanical properties and examples of material systems.

Type of ceram and Classifications		Flexural strength (MPa) *	Fracture toughness (MPa m ^{1/2})*	Example of material systems
Feldspathic porcelain	Porcelain	50-120	~ 1.0	Duceram® Plus
Leucite-reinforced glass ceramic	Glass ceramic	120-180	~ 1.5	IPS Empress®
Lithium-disilicate reinforced glass ceramic		350- 400	~ 3.0	IPS e.max® Press IPS e.max® CAD
Glass-infiltrated aluminum oxide	Hybrid ceramic	450-500	3.5-4.0	In-Ceram® Alumina
Glass-infiltrated zirconia-toughened aluminum oxide		650-700	4.5-5.0	In-Ceram® Zirconia
Densely sintered aluminum oxide	Oxide ceramic	420-650	3.0-4.0	Procera® Alumina
Y-TZP		900-1200	6.0-8.0	Procera® Zirconia Denzir®

Y-TZP= Yttrium oxide-stabilized tetragonal zirconium dioxide polycrystals

*from: Vult von Steyern P. Dental ceramics in clinical practice. In: Nilner K, Karlsson S, Dahl B L (ed). A textbook of fixed prosthodontics: the Scandinavian approach. Stockholm: Gothia 2013: 205-222 (13). Rekow E.D et al. Performance of dental ceramics: challenges for improvements J. Dent. Res.2011;90: 937-952 (26). Miyazaki T. et al. Current status of zirconia restoration. J. Prosthodont. Res. 2013;57:236-261 (5).

The hybrid ceramics consists of glass-infiltrated aluminum oxide (Al₂O₃, alumina) or glass-infiltrated zirconium dioxide- (ZrO₂, zirconia) toughened alumina (4). Hybrid ceramics have mainly been used for single crowns and FDPs with up to three units as substructures, veneered with porcelain (8, 27). Use of this ceramic group has decreased due to their technique sensitive and time-consuming fabrication process and the increased use of lithium disilicate glass ceramics and oxide ceramics (4).

Oxide ceramics, often defined as polycrystalline, high-strength oxide ceramics, lack a glass phase and are acid resistant. Densely sintered aluminum oxide (high purity alumina) and Y-TZP with no glass phase are the two main oxide ceramics in dentistry (24, 28, 29).

Due to its mechanical properties, Y-TZP has a wide range of uses; crowns, FPDs, posts, and implant abutments (11, 30-32). Y-TZP is used for substructures, with veneer material consisting of porcelain or glass ceramics. In many cases it has replaced dental appliances in aluminum oxide (4). Aluminum oxide is still used, but mainly as a substructure for single crowns. Since the substructure, i.e. the oxide ceramic, is what determines the strength, the restorations do not need to be adhesively cemented if mechanical retention is provided (24, 32-34). Research is constantly developing stabilized zirconia materials to improve its clinical use and outcomes, and to meet future demands. Recent developments include translucent Y-TZP, allowing monolithic crowns to be made that maintain the favorable mechanical properties of Y-TZP (35).

Aluminum oxide and stabilized zirconium dioxide

Densely sintered aluminum oxide consists of high purity (99.9%) aluminum oxide. The aluminum oxide powder is pressed and sintered (4, 36). During the sintering the aluminum oxide particles grow together to a grain-like structure, having a mean grain size of 4 μm . The sintering shrinkage of densely sintered aluminum oxide is approximately 20%, which is controlled during the manufacture of an individual substructure (36-38).

Unlike aluminum oxide, zirconium dioxide is a polymorphic material that shows three different crystal structures, depending on the temperature. From room temperature (RT) up to approximately 1170°C the crystals have a monoclinic structure. Above 1170°C, phase transformation occurs and the monoclinic crystals become tetragonal. At about 2370°C, the crystals have a cubic structure. And if the temperature is further raised, the material will melt at around 2680 °C. During cooling, reversed transformation occurs back to tetragonal and then monoclinic structure, increasing the crystal size (volume) with approximately 3% to 5%. This increase causes undesirable concentrations of stress and, sometimes, spontaneous crack formation within the material at RT. Hence, pure zirconium dioxide is unsuitable for dental use. By adding a stabilizing oxide, a dopant, the tetragonal phase can be stabilized at RT. Examples of such dopants are yttrium (Y_2O_3), magnesium (MgO), cerium (Ce_2O_3) and calcium (CaO) oxide. In dental contexts, zirconium dioxide is

most frequently stabilized with 3 mol% yttrium oxide, (3Y-TZP). The Y-TZP will be metastable, i.e. the tetragonal zirconium dioxide is stabilized at RT but can transfer to monoclinic structure under local stress (4, 29, 31, 39, 40).

Flaws and cracks play an important role in the fracture mechanism of dental ceramics. Fractures often originate from such defects, which act as starting points for slow crack growth (41). When a crack starts to propagate in the Y-TZP, a local stress-initiated phase transformation will occur at the crack tip. This transformation from tetragonal to monoclinic phase causes a local increase in volume of the crystals (approximately 3%) close to the tip of the crack, resulting in a localized compressive stress that will prevent or delay further crack propagation. This mechanism is known as transformation toughening, and it is the underlying reason for the high fracture toughness of Y-TZP. However, this metastability means that Y-TZP is likely to age in the presence of water, hot vapor, steam sterilization, body fluids like saliva, or mechanical treatments such as grinding or airborne particle abrasion (39, 42-44). Water can “catalyze” stress corrosion, breaking the bond between the atoms at the crack tip, leading to slow crack growth where the transformation of tetragonal to monoclinic phase takes place. The process starts at the surface grain boundaries and the transformation continues layer by layer through the whole body, resulting in microcracks, grain pullout, and decreased strength. This degradation or aging of Y-TZP is also known as low temperature degradation (LTD), which is defined as the spontaneous transformation from tetragonal to monoclinic at low temperature over time. Y-TZP is prone to LTD in the presence of water (43, 44).

Y-TZP is less translucent than other dental ceramic materials, so development has focused on increasing its translucency (5, 35). The translucency of stabilized zirconium dioxide is related to the amount and type of dopants; the amount and size of crystals and pores; and the sintering process, including the sintering temperature, heat rating, and atmosphere during the sintering process (45, 46). Few clinical studies of translucent Y-TZP are available, however, and further investigations are needed to evaluate the performance (5, 35).

Process technology

The properties of oxide ceramics largely depend on its processing, including the ceramic powder used, the fabrication technique, and final treatment of the restoration. The powder should be as pure as possible and the particle size sufficiently small to allow the final packing and sintering that creates optimal mechanical properties (39, 40, 47, 48).

Oxide ceramics for dental appliances are commonly produced by pressing ceramic granules into a body. These are subsequently sintered and milled by computer-aided design/computer-aided manufacturing (CAD/CAM). CAD/CAM techniques involve producing a digital model of the dental arches, the prepared tooth/teeth, and designing a restoration using the CAD-software (31, 49).

There are three compaction techniques used to increase the density of the ceramic granules: uniaxial pressing, cold isostatic pressing (CIP), and hot isostatic pressing (HIP). In the uniaxial press technique, the pressure is applied in one direction, yielding a green body with low material pack, especially in the outer contours due to friction against the walls of the mold. The material will have a somewhat lower density and may have inherent stresses (50, 51). Isostatic press techniques make the material more homogenously packed because pressure is applied in all directions. This creates higher density, fewer pores and voids, and isotropic properties (50). There are two methods for CIP, wet bag and dry bag. In wet bag process, the powder is enclosed in a waterproof mold with flexible walls and then placed in a container filled with liquid and put under pressure. Dry bag, however, is performed with very little liquid. The mold is connected to channels filled with pressurized liquid that compress the material. HIP includes isostatic pressing with pre-sintering, followed by a temperature increase to between 1400°C and 1500°C under high pressure in an inert gas atmosphere. This creates a very dense material (99% of the theoretical density) (50). Y-TZP produced by HIP has been reported to be less sensitive to LTD (32). However, the milling procedure will affect the surface and the crystalline structure, causing some degree of tetragonal to monoclinic phase transformation in the Y-TZP (31).

With stabilized zirconium dioxide, milling is performed either by soft machining the oxide ceramic in its green stage (pressed only) or

white stage (pressed, partially sintered), or by hard machining the fully sintered stage (pressed and completely sintered). When a restoration is milled from a blank, the production technique is subtractive. When the restoration is built up by adding ceramic granules to achieve the desired design, the production technique is additive (31, 49). The two techniques can be combined by pressing the ceramic granules on an enlarged die and then subsequently milling the outer contours to the desired shape (36, 37). The machining process of subtractive production leaves a certain structural roughness in the surface, depending on the milling tools used (11, 31, 52). With additive production, however, the ceramic granules are pressed to green stage on the surface of a prefabricated die. Subsequently, the machining process shapes the outer contours whereby the surface structure of the cementation surface mirrors the surface structure of the die, which is often quite smooth (36, 37).

Clinical use and issues

The mechanical properties of oxide ceramics allow for cementation of crowns and FDPs with conventional cements, such as zinc phosphate cement (24, 32-34). However, in some clinical cases, such as those with compromised retention, it might be beneficial to apply adhesive cementation and establish a durable bond between the restoration and the tooth substance (34). Some restorations, such as resin-bonded bridges which are commonly made of porcelain fused to metal (PFM), rely on adhesive cementation. Disadvantages of metal-framed, resin-bonded bridges include decreased translucency and a greyish appearance of the abutment teeth. With resin-bonded all-ceramic bridges (RBCBs) the same minimally invasive approach can be applied. RBCBs, on the other hand, do not have the same strength as the metal-framed and may have higher risk of fracture. Oxide ceramics, with well-dimensioned design and beneficial mechanical properties compared to other ceramics, might not encounter this issue. Longitudinal clinical studies of oxide-based RBCBs are lacking (53, 54).

Compared to silica-based ceramics, which can be bonded after HF etching and silanization (24), densely sintered oxide ceramics have surface structures without glass phase. They require alternative techniques for adhesive cementation (33, 55). The reliability

of the long-term bond to oxide ceramics is determined by the micromechanical and chemical retention between the adhesive cement system and the surface of the ceramic restorations (24, 55-57). Macromechanical retention, however, is important to avoid unacceptably high stress levels that may affect the interfacial bond between restoration/cement/tooth, potentially reducing the clinical performance of the restoration (9).

Micromechanical retention is determined by the structure of the restoration's cementation surface. The cementation surface will differ in surface roughness, depending on the manufacturing technique, thereby influencing bond strength (20, 48, 58-61). With a rougher surface, the size of the surface area will increase and, in turn, affect wettability. This allows the cement to flow into the microretentions, thus creating a stronger micromechanical interlock (61, 62).

The literature describes several methods of surface treatment and modification (33, 55). These can be divided into: (i) abrasive techniques, such as grinding/polishing and airborne particle abrasion also known as sandblasting (20, 63-65) and (ii) additive surface treatments, such as tribochemical silicon dioxide (silica) coating (64,66-68), silanization (64), plasma spraying, (69) fusion sputtering, (70) laser irradiation, (71-73) selective infiltration etching, (74, 75) ceramic powder (67-69, 76, 77) or nanostructured alumina coating, (78) and primers (55, 66, 79-81). Some of these techniques, however, are not yet commercially available (33). There are some drawbacks to the various surface treatments. Different surface modification techniques e.g. abrasive techniques can result in structural damage, such as grain pullout and material loss, creating flaws that can decrease strength (57, 76, 82-85). Other coating techniques do not induce any surface flaws, but might instead affect the fit of a restoration as the coating adds material to the surface (69, 86).

To promote chemical retention, different cement systems have been proposed for adhesive cementation of oxide ceramics in an attempt to attain durable bonding to the ceramic restoration (5, 55). Hence, silica/silane bonds are not the only way to achieve a stable bond. Several primers with reactive monomers have been evaluated *in vitro* in order to investigate their bond to oxide ceramics (11, 52, 87-91). The monomer 10-methacryloyloxy-decyl dihydrogenphosphate

(MDP) was originally designed to bond to metal oxides, and its use has been extended to oxide ceramics (92). MDP-containing resin cements seem to be the most appropriate for oxide ceramics due to the chemical interaction between the hydroxyl groups of the passive zirconium dioxide surface and the phosphate ester group of the MDP. Some suggest that a chemical bond might be established between MDP and oxide ceramics (20, 34, 56, 57, 64). Regular bisphenol-A-diglycidyl-methacrylate (bis-GMA) resin cements, which do not contain MDP, might improve the bond strength to aluminum oxide and stabilized zirconium dioxide if combined with an MDP-containing primer (55, 87). Chemical retention alone is difficult and unpredictable to obtain for oxide ceramics (52, 74, 78, 91, 93). Several studies have concluded that surface treatment that creates micromechanical retention is beneficial to chemical retention, thereby allowing a durable bond between oxide ceramics and adhesive cement systems (34, 57, 91, 94, 95). Nevertheless, the literature indicates that establishing a strong and reliable bond between oxide ceramics, particularly Y-TZP, and a bonding component is both difficult and unpredictable (9, 24, 55).

What do we know today? – Final remark

Achieving long-term bond strength of oxide ceramics to adhesive cement systems could result in wider applications for oxide-based ceramic restorations, especially those that rely heavily on bonding. By using adhesive cementation, it would also be possible to decrease the need for excessive tooth preparation, thus preserving tooth substance. However, there is no conclusive evidence or consensus regarding the most suitable materials and techniques for creating a durable bond to oxide ceramics. Hence, further studies are needed to investigate different surface treatments for oxide ceramics and their effect on bond strength and material properties.

HYPOTHESES

- There is no difference between the shear bond strength achieved on densely sintered aluminum oxide with different adhesive cement systems or with different surfaces treatments.
- A modified-additive impaction technique can achieve a bondable surface on Y-TZP.
- The surface structure of surface-modified Y-TZP will differ from an unmodified surface of Y-TZP. Surface roughness will increase when granules are added to the surface.
- The chemical surface composition of surface-modified Y-TZP will differ from an unmodified surface of Y-TZP. Glass phase remnants and various phases will be present after sintering on the surface of the Y-TZP produced with modified-additive production.
- There is no difference for either unmodified or surface-modified Y-TZP between the shear bond strength achieved with different adhesive cement systems.
- There is no difference in flexural strength, regardless of surface modification or pretreatments, either with or without an adhesive cement system.

AIMS

The overall aim of this thesis was to evaluate and develop techniques for modifying the surface of oxide ceramics that enable durable bonding between the restorations and adhesive cement systems. Additionally, the thesis inventories existing methods for achieving a bondable surface on oxide ceramics and how these methods affect the materials.

Specific aims

- Evaluate whether sufficient and durable shear bond strength (>20 MPa) can be established between various adhesive cement systems and densely sintered aluminum oxide (as-produced or sandblasted).
- Describe a method for producing a bondable Y-TZP surface using impaction modification with two different mediums: glass granules and polymer granules.
- Investigate and describe the surface structures of surface-modified Y-TZP produced using modified-additive technique and compare them to an unmodified Y-TZP surface.
- Investigate and describe the chemical surface composition of surface-modified Y-TZP produced using modified-additive technique and compare them to an unmodified Y-TZP surface.
- Evaluate the shear bond strength (>20 MPa) between various adhesive cement systems and both unmodified and surface-modified Y-TZP.

- Evaluate the flexural strength of Y-TZP, with surface modification and without, with various pretreatments (etching either before or after sintering, both with an adhesive cement system and without).
- Make an inventory of existing methods for achieving a bondable surface on oxide ceramics and evaluate which methods might provide sufficient bond strength.

MATERIALS AND METHODS

This thesis comprises three *in vitro* studies and a systematic review. Table 2 summarizes the various materials and methods that are explained in this section. For further details, see the “Materials and methods” sections of each individual study.

Laboratory Procedures

Studies I, II and IV

These studies evaluated the bond and flexural strength of the oxide ceramics aluminum oxide and stabilized zirconium dioxide with surface treatment of the cementation surface and various adhesive cement systems.

Study I

Specimen preparations

Study I used 120 pairs of industrially manufactured specimens – one block and one cylinder (Ø 6.0 mm, thickness of 2.00 mm) of densely sintered aluminum oxide (Procera® Alumina, Nobel Biocare AB, Gothenburg, Sweden). The specimens were divided into 12 groups (n=10) depending on the adhesive cement system used (six different systems were tested) and on the artificial aging procedure (water storage only or storage including thermocycling [TC]).

Preparation of the cementation surface of the blocks involved cleaning the blocks with acetone (Apoteksbolaget AB, Gothenburg, Sweden) and then performing airborne particle abrasion with 110 µm aluminum oxide particles (Aloxcobra, Renfert GmbH, Hilzingen, Germany) for 10 seconds at an air pressure of 5 bars and a distance of 100 mm between the surface of the sample and the

Table 2. Summary of materials and methods.

Paper	I		II		III		IV	
	Type	<i>In vitro</i>	<i>In vitro</i>	Literature review	<i>In vitro</i>		<i>In vitro</i>	
Specimens/ Studies	120 blocks + cylinders	48 blocks + cylinders		127 studies for Q1 23 studies for Q2			248 discs	
Oxide ceramic	Procera® Alumina	Unmodified Y-TZP, Glass- or polymer-modified Y-TZP		12 brands of Y-TZP 2 brands of Alumina			Unmodified Y-TZP, Glass-modified Y-TZP	
Adhesive cement system	Variolink® II BISCO CHOICE™ BISCO ILLUSION™ NEXUS 2™ 3M™ ESPE™ RelyX™ Veneer Cement Panavia™ F	Variolink® II Panavia™ F 2.0		*			Panavia™ F 2.0	
Artificial aging	Water storage (1 week in 37° C water) or including TC (5 000 cycles 5° C to 55° C)	TC (5 000 cycles 5° C to 55° C)		Storage of test specimens, or other methods with mechanical cyclic loading (>500 cycles) in combination with water/liquid			Preloading (10,000 cycles, 10 N to 100 N) and TC (5 000 cycles 5° C to 55° C)	
Test method	Shear bond strength test	Shear bond strength test		All test methods for bond strength without the influence of macromechanical retention			Biaxial flexural strength test	
Surface analysis	Light microscopy	Light microscopy, IFM		*			Light microscopy, IFM, AFM, SEM, EDS, XRD	
Statistical method	Student's t-test, one-way ANOVA, Tukey's test and Fisher's exact test	One-way and two-way ANOVA, Tukey's test, and Fisher's exact test		*			One-way ANOVA, Tukey's test	

* not evaluated, Q= addressed question in the literature search, TC= Thermocycling, IFM= Interferometry, AFM= Atomic force microscopy, SEM= Scanning electron microscopy, EDS= Energy-dispersive X-ray spectroscopy, XRD= X-ray diffraction.

blasting nozzle. The nozzle of the blaster was moved gently in circles at a 70° angle to the surface. After sandblasting, the samples were ultrasonically cleaned in 96% isopropanol (Apoteksbolaget AB, Gothenburg, Sweden) for 5 minutes. The cylinder-shaped samples were not sandblasted, but stored as-received from the manufacturer and subsequently cleaned, first with acetone and then ultrasonically in 96% isopropanol for 5 minutes.

Study I tested six different adhesive cement systems. Table 3 lists the various systems and their characteristics. Prior to cementation all cementation surfaces were treated, irrespective of group, with the primer recommended for each adhesive cement system by the cement's manufacturer. The untreated cylinders were cemented to the prepared blocks using an alignment that applied a seating load at 15 newtons (N) during polymerization. The apparatus standardized the seating load and ensured that the axes of the cylinder were perpendicular to the surface of the block. Disposable brushes (Top Dent, DAB Dental AB, Sweden) were then used to remove excess resin from the margin. If it was part of the adhesive cement system, an oxygen-blocking gel was applied according to the manufacturer's instructions (Table 3). The cement were light-cured with dental curing lamp (Optilux 400, Model VCL 401, Demetron Research Corporation, Danbury, CT, USA) with a mean light intensity of 300 mW/cm². The curing time was 40 seconds from four directions, 90° apart, and then 60 seconds in one direction with the seating load removed. All excess resin was removed with a surgical blade (AESCULAP® no. 12, Aesculap AG & Co, Tuttlingen, Germany) after polymerization. In a final step, the specimens were rinsed with water for 1 minute to remove any residues of the oxygen-blocking gel.

Each bonding group of 20 samples was randomly divided into two subgroups (n=10) for further treatment: water storage only or storage including TC. Both subgroups were stored in 37°C distilled water for 1 week. During this week, the subgroups that included TC received further artificial aging.

Table 3. Adhesive cement systems used in Study I.

System and components	Abbreviation	Type	Manufacturer	Batch No./LOT
VARIOLINK® II	VA	Bis-GMA based resin*	Ivoclar Vivadent AG/ FL-9494 Schaan/ Lichtenstein	
Monobond-S, primer agent		Dual-cured		D511336
Heliobond, porcelain primer		Two-paste system		D68417
Varolink® II, Base + Catalyst, (T)				D20542 + D17352
Liquid Strip				D50843
<hr/>				
BISCO CHOICE™	BC	Bis-GMA based resin	Bisco Inc/ Schaumburg, IL 60193/ USA	
BISCO PORCELAIN PRIMER		Dual-cured		0200007636
ONE-STEP® Univ. dental adhesive		Two-paste system		0100012867
CHOICE™ Porcelain Adhesive				0100014106 +
Paste Base (translucent) + Catalyst				0100014108
<hr/>				
BISCO ILLUSION™	BI	Bis-GMA based resin	Bisco Inc/ Schaumburg, IL 60193/ USA	
BISCO PORCELAIN PRIMER		Dual-cured		0200007636
ONE-STEP® Univ. dental adhesive		Two-paste system		0100012867
ILLUSION™ Porcelain Adhesive				0100014248 +
Paste Clear Base				0100013914
+ Clear Catalyst				

(Table 3 continues on next page)

(Table 3 cont.)

NEXUS 2™ Silane Primer	NE	Bis-GMA based resin Dual-cured	Kerr Corporation/ Orange, CA 92867/ USA	010381
NEXUS 2™ Base Clear + Catalyst		Two-paste system		010435 + 010440
3M™ ESPE™ RelyX™ Veneer Cement	RV	TEGDMA** / Bis-GMA based resin	3M ESPE/ St. Paul, MN 55144/ USA	
3M™ RelyX™ Ceramic Primer		Light-cured		20020716
3M™ Scotchbond™ 1, adhesive		One-paste system		20020620
3M™ ESPE™ RelyX™ Veneer Cement (TR)				20011019
PANAVIA™ F	PF	Phosphatemonomer containing resin (MDP)***	KURARAY MEDICAL INC/ Okayama 710- 8622/ Japan	0563B
CLEARFIL™ PORCELAIN SE BOND ACTIVATOR				
CLEARFIL™ SE BOND PRIMER		Dual-cured		00120B
PANAVIA™ F A Paste + B Paste (TC)		Two-paste system		00050A + 00029A
PANAVIA™ F OXYGUARD II				00433A

* Bis-GMA= Bisphenol-A-diglycidylmethacrylate, **TEGDMA =Triethilene glycol-dimethacrylate, ***MDP = 10-methacryloyloxydecyl-dihydrogenphosphate

Artificial ageing – TC

All specimens underwent TC at 5 000 cycles in a specially constructed thermocycling device with two water baths: one at 5°C and the other at 55°C. Each cycle lasted 60 seconds (20 seconds in each bath and 10 seconds for transfer between the baths).

Bond strength test

Following pretreatment and artificial aging, a universal testing machine (Instron model 4465, Instron®, Canton, MA, USA) measured the shear bond strength of the samples using a knife-edged blade parallel to the bonded surfaces, in accordance with ISO/TS 11405:2003 Dental materials –Testing of adhesion to tooth structure (96) and previous studies (63, 97). The blocks were placed in a brass holder fixed to the testing device to maintain their position during testing. The crosshead speed was 0.5 mm/minute. The load at fracture was recorded in newton (N). The shear bond strength in megapascals (MPa) was calculated by dividing the recorded load by the area of the cementation surface, measured individually in mm², and determined the mean and standard deviation for each group.

Failure type

The fracture surfaces were examined in a light microscop (Wild M3, Wild Heerbrugg, Heerbrugg, Switzerland at ×6.4 magnification) to classify the type of failure of the debonded area: adhesive, cohesive, or a combination of the two.

Surface analysis and characterization

Microscopy. During all steps in the fabrication process of the specimens, two types of light microscope were used to analyze the cementation surfaces and fracture surfaces (Wild M3, Wild Heerbrugg, Heerbrugg, Switzerland at ×5 to ×10 magnification and Leica DM 2500M, Leica Microsystems CMS, Wetzlar, Germany at ×500 magnification).

Statistics

Student's t-test and one-way ANOVA, Tukey's test determined differences in bond strength within and between the groups (IBM SPSS Statistics 15.0, SPSS Inc., Chicago, IL, USA). The level of significance was set to $\alpha = 0.05$.

Study II

Specimen preparations

In this study, 48 pairs of specimens were fabricated: one Y-TZP cylinder and one block made of feldspathic porcelain that was adhesively cemented together. The specimens were divided into six groups (n=8/group) depending on the cementation surface of the Y-TZP cylinder and adhesive cement system (three different surfaces and two different systems).

A special dry-press punching tool made of stainless steel was made to fabricate the Y-TZP cylinders. To fabricate the specimens, the tool was filled with 0.33 ± 0.01 g of Y-TZP granulated powder (grade TZ-3YSB-C, batch S306269B, Procera Zirconia, Nobel Biocare™ AB, Gothenburg, Sweden) mechanically compressed uniaxially with 125 MPa using a cuvette press. Prior to compaction, the bottom punch surfaces of the pressing tool (which define the cementation surface of the Y-TZP) was modified by applying a thin layer of one of two different mediums onto the surface (98). The three groups varied depending on their surface modifications:

- Surface G: Glass granules (Experimental Impaction Medium, Cerasci AB, Malmö, Sweden) with a particle size of 40 μm or less covering the bottom punch surface prior to compaction.
- Surface P: Polymer granules (Cystrip U type 2 20/30, Blästerprodukter Köping AB, Köping, Sweden) with a particle size of 40 μm or less covering the bottom punch surface prior to compaction.
- Control (C). No medium was added to the bottom punch prior to compaction.

The tool was cleaned with 95% ethanol prior to each compaction. The cylinders, regardless of group, were sintered in a sintering furnace (Everest Therm 4180, KaVo Everest, Biberach®, Germany) according to the manufacturer's instructions. The cylinders were measured after sintering, which ranged in diameter from $\text{Ø } 4.97$ to 5.11 mm, with a height of $3 \text{ mm} \pm 0.1$ mm. Measurement was made using a digital caliper (Powerfix® Electronic digital caliper, Paget Trading, London, UK).

Feldspathic porcelain blocks were manufactured by using porcelain (Duceram® Plus dentin A3.5, Degudent, Hanau-Wolfgang, Germany) shaped and fired in a calibrated porcelain furnace (Programat P500, Ivoclar-Vivadent, Schaan, Liechtenstein) using one dentin and one self-glaze firing, with all steps performed according to the manufacturer's recommendations.

The cementation surfaces of both the porcelain blocks and Y-TZP cylinders were treated with 9.6% HF (Top Dent 9.6%, DAB Dental, Upplands Väsby, Sweden), thoroughly rinsed with water, cleaned with 35% phosphoric acid (Ultra-Etch 35%, Ultradent Products, South Jordan, UT, USA) and again thoroughly rinsed with water. After manufacturing, the specimens (n=8/group) were randomly divided into subgroups depending on the adhesive cement system used: either Variolink®II (Ivoclar-Vivadent AG, FL-9494 Schaan, Liechtenstein) or Panavia™F 2.0 (Kuraray Medical, Osaka, Japan). Prior to cementation, all cementation surfaces were treated with a primer appropriate to the adhesive cement system, according to the cement manufacturer's recommendations. Study II used the same cementation procedures as Study I, apart from the light-curing. Light-curing in Study II used a different curing lamp (Ivoclar-Vivadent Bluephase, Scaan, Liechtenstein). Polymerization light intensity was 1100 mW/cm² and curing time was 20 seconds in each of four directions, 90° apart, and then 60 seconds in one direction with the seating load removed. All specimens were stored for 10 hours in a humid environment to avoid desiccation during storage before the additional treatment of artificial aging.

Artificial ageing – TC

All specimens were thermocycled at 5 000 cycles in the same manner as Study I.

Bond strength test

Following pretreatment and artificial aging, a universal testing machine (Instron model 4465, Instron®, Canton, MA, USA) measured shear bond strength using a knife-edged blade parallel to the bonded surfaces as in Study I. From the data collected the mean and standard deviation for each group were calculated.

Failure type

The fracture surfaces were examined in a light microscope (Wild M3, Wild Heerbrugg, Heerbrugg, Switzerland at $\times 6.4$ magnification) to classify the type of failure in the debonded area: adhesive, cohesive, or a combination of the two.

Surface analysis and characterization

Microscopy. Two types of a light microscope were used to analyze the cementation surfaces and fracture surfaces (Wild M3, Wild Heerbrugg, Heerbrugg, Switzerland at $\times 5$ to $\times 10$ magnification and Leica DM 2500M, Leica Microsystems CMS, Wetzlar, Germany at $\times 500$ magnification).

Interferometry. The samples were examined with interferometry (IFM) using a MicroXAM™ instrument (ADE Phase shift Technology, Inc., Tucson, USA), in order to characterize the surface roughness at the micrometer level. The IFM had a maximum resolution of 0.05 nm in the vertical direction and 0.3 μm in the lateral direction. The scanned area of the specimens was 200 \times 260 μm . The images obtained by IFM were subjected to leveling and Gaussian filtering (size 50 μm \times 50 μm), and the roughness parameters were calculated using the software MapVue (MetaMAP, Lexington, KY, USA). Three parameters were selected according to proposed guidelines for biomaterial surface characterization (99): one height descriptive, S_a = the arithmetic average height deviation from mean plane (μm), one spatial descriptive, S_{ds} = the density of summits (μm^{-2}), and one hybrid parameter S_{dr} = the developed surface ratio (%). Three specimens from each group and three measurements per specimen ($n=9/\text{group}$) were made.

Statistics

One-way ANOVA, Tukey's test (IBM SPSS Statistics 17.0, SPSS Inc., Chicago, IL, USA) determined differences in bond strength between the groups. Two-way ANOVA, Tukey's test provided statistical analysis of the surface roughness between groups. Fisher's exact test tracked differences in the type of failure between each group. The level of significance was set to $\alpha = 0.05$.

Study IV

Specimen preparations

Study IV performed surface analysis (n=48) and a biaxial strength test (n=200 on Y-TZP discs. The specimens were divided into groups depending both on the cementation surface of Y-TZP (either unmodified Y-TZP [C], or glass-modified Y-TZP surfaces [G]) and on the production process (either etching before sintering [CE/GE], sintering [CS/GS], sintering followed by etching [CSE/GSE], sintering followed by sandblasting [CSS], sintering and sandblasting followed by etching [CSSE]) all tested with or without the use of cement, Panavia™F 2.0 (Kuraray Medical, Osaka, Japan) Table 4.

The method of specimen fabrication was the same as for Study II. The tool was filled with 0.75 -1.15 g \pm 0.01 g of Y-TZP granulated powder that was mechanically compressed uniaxially with 140 MPa for five minutes, using a cuvette press. The bottom punch surface of the pressing tool, which defines the cementation surface of the Y-TZP, had either no medium added prior to compaction (C) or glass granules with a particle size of 40 μ m or less (G), Table 4. The tool was cleaned with 95% ethanol (Ethanol 95%, batch nr. SE10016023, Kemetyl AB, Haninge, Sweden) before each compaction.

Prior to sintering, the specimens with unmodified surface (CE) and those with glass-modified surface (GE, GEC,) were etched for two minutes using 9% HF. Then the specimens were neutralized for two minutes before rinsing them thoroughly with water and storing them at room temperature 24 hours. After this, all specimens, regardless of group, were sintered in a sintering furnace according to the manufacturer's instructions. After sintering, specimens with unmodified surface (CS) either underwent no further treatment or they were sandblasted with 110 μ m Al₂O₃ particles for 10 seconds with an air pressure of 2 bars at a distance of 10 mm with gentle movements of the blasting nozzle perpendicular to the surface before being thoroughly rinsed with water (CSS, CSSC). The remaining specimens with the unmodified surface (CSE), sandblasted surface (CSSE) and with the glass-modified surface (GSE, GSEC) were etched with 9% HF according to the etching procedure described in Table 4.

Table 4. Overview of the groups, materials, pretreatments used and the analysis performed.

Groups (Abbreviation)	Materials used	Pretreatment/Sintering	Surface analysis (n=6)	Flexural strength test (n=25)
CE	Y-TZP* + HF#	Etched [#] before sintered ^{**}	IFM, AFM, SEM, EDS	-
CS	Y-TZP	Sintered	IFM, AFM, SEM, EDS	Yes
CSE	Y-TZP + HF	Sintered + etched	IFM, AFM, SEM, EDS	-
CSC	Y-TZP + PanaviaF TM 2.0 ^{##}	Sintered + cement ^{###}	-	Yes
CSS	Y-TZP + Al ₂ O ₃ 100µm ^{***}	Sintered + sand-blasted ^{***}	IFM, AFM, SEM, EDS	Yes
CSSE	Y-TZP + Al ₂ O ₃ 100µm+ HF	Sintered + sandblasted + etched	IFM, AFM, SEM, EDS	-
CSSC	Y-TZP + Al ₂ O ₃ 100µm + PanaviaF TM 2.0	Sintered + sandblasted + cement	-	Yes
GE	Glass-modified Y-TZP ^{###} + HF	Etched before sintered	IFM, AFM, SEM, EDS	Yes
GEC	Glass-modified Y-TZP + HF + PanaviaF TM 2.0	Etched + sintered + cement	-	Yes
GS	Glass-modified Y-TZP	Sintered	IFM, AFM, SEM, EDS	-
GSE	Glass-modified Y-TZP + HF	Sintered + etched	IFM, AFM, SEM, EDS	Yes
GSEC	Glass-modified Y-TZP + HF + PanaviaF TM 2.0	Sintered + etched + cement	-	Yes

* Y-TZP: grade TZ-3YSB-C, (batch S306269B, Procera Zirconia, Nobel BiocareTM AB, Gothenburg, Sweden). # HF: etched with 9% hydrofluoric acid (Ultradent[®] Porcelain Etch, 9%, LOT B7814, Ultradent Products, Inc., South Jordan, Utah, USA) for two minutes, neutralized (IPS Ceramic, Neutralizing powder, LOT M04796 Ivoclar Vivadent, Schaan, Liechtenstein) for two minutes, followed by thorough rinsing with water according to the cement manufacturer's recommendation. ** Sintered: sintered in a sintering furnace (Everest Therm 4180, KaVo Everest[®], Biberach, Germany) according to the manufacturer's instructions. ## PanaviaFTM2.0: treated with a primer (Clearfil Ceramic Primer, LOT 0025AA, Kuraray Noritake Dental Inc., Okayama, Japan) prior to application of the adhesive cement, PanaviaTMF 2.0 (PanaviaTMF 2.0, base paste LOT 0112AA and catalyst paste LOT 0575AA, Kuraray Noritake Inc., Okayama, Japan) and oxygen-blocking gel (PanaviaTMF 2.0 Oxyguard II, LOT 00655A, Kuraray Medical Inc., Okayama, Japan) according to the cement manufacturer's recommendation. *** Al₂O₃ 100 µm: sandblasted with 110 µm Al₂O₃ particles for 10 seconds with an air pressure of 2 bar and at a distance of 10 mm, then thoroughly rinsed in water. ### Glass-modified YTZP: modified with glass granules (Experimental Impaction Medium, Cerasci AB, Malmö, Sweden).

Based on a power analysis consisting of a two-sample test with a significance level of 5% with true difference of means set at 50 N and with a power of 95%, 200 specimens of Y-TZP were prepared for the biaxial test. This included a total of four subgroups, each with or without an adhesive cement system, Panavia^{TMF} 2.0 (n=25), Table 4. The fabrication process was the same as for the fabrication of the specimens for surface analysis and the final size of the discs, Ø 12.8±0.2 mm and a height of 1.29±0.1 mm, accorded with ISO 6872:2008 (18). All discs were subjected to heat treatment to simulate the firing cycles of a recommended veneering porcelain (GC Initial ZR-FS, GC Europe N.V., Leuven, Belgium). Firing cycles proceeded in a calibrated furnace and each disc underwent four firings: frame modifier, dentin 1, dentin 2 and self-glaze firing, all according to the manufacturer's recommendations.

All subgroups with the surface covered by cement (CSC, CSSC, GEC, GSEC) were etched with HF, according to the same etching procedure described above; etching prior sintering. All these discs were treated with a primer before application of the adhesive cement Panavia^{TMF} 2.0 according to the cement manufacturer's recommendation, Table 4. The cement was applied to the cementation surface of the Y-TZP and a 0.12 mm thick plastic film was placed between an alignment apparatus with a seating load of 15 N during polymerization. The excess resin was removed from the margin using disposable brushes and the cement was light-cured with a curing lamp (Heraeus Translux[®] Power Blue[®], Hereaus Kulzer GmbH, Hanau, Germany). The polymerization light intensity was 1000 mW/cm² and the curing time was 20 seconds for each of four directions, 90° apart, and then 60 seconds in one direction with the seating load removed. Subsequently, an oxygen-blocking gel was applied for 3 minutes, followed by a thorough rinse with water for 1 minute to remove any residue of the oxygen-blocking gel, Table 4. After polymerization, all the excess of resin was removed with a surgical blade and then stored the discs in water at RT (22°C) for 24 hours to avoid desiccation during storage.

Artificial ageing – Cyclic preload and TC

Before the biaxial flexure strength test, all specimens underwent artificial ageing with cyclic preloading and TC. The specimens were subjected to cyclic preload at loads between 10 and 100 N at 1 Hz in a wet environment for 10,000 cycles using a specially constructed preloading device (MTI Engineering AB, Lund, Sweden/Pamaco AB, Malmö, Sweden). The discs were placed on three supporting steel balls (\varnothing 2.5 mm) while a centrally placed stainless punch (\varnothing 1.4 mm) applied the load at the center, perpendicular to the discs. A 0.12 mm thick plastic film was placed both between the three supporting balls and the disc, and between the punch and the disc. Subsequently, all specimens were thermocycled at 5 000 cycles in two baths; one at 5°C and the other at 55°C, as described previously in Studies I and II.

Biaxial flexural strength

The specimens were placed in a universal testing machine (Instron model 4465, Instron®, Canton, MA, USA) according to the cyclic preloading, and loaded to the point of fracture in a wet environment. The crosshead speed was 0.5 mm/min. Load at fracture (N) was registered when a visible fracture occurred. The flexural strength in MPa was calculated according to ISO 6872:2008 Dentistry – Ceramic materials (18) and with the Poisson's ratio of 0.25. Throughout the test period, whenever the specimens were not being actively tested, they were stored in a humid environment at RT.

Surface analysis and characterization

Microscopy. During all steps in the fabrication process of the specimens, the cementation surfaces were analyzed under light microscopes, as described in Study II.

Interferometry. To define and characterize the surface, IFM, was used as described in Study II. Three specimens from each group and three measurements per specimens (n=9/group) were analyzed, Table 4.

Atomic force microscopy. To further characterize the surface, atomic force microscopy AFM analysis (XE-100, Park Systems, Suwon, Korea) was performed in intermittent-contact mode using etched silicon probes with cantilever lengths of 125 nm and nominal resonance frequencies of 270-310 KHz. A scanning area of 10 x 10 μm with a resolution of atomic level in the vertical direction and 2 nm in the lateral direction was used and the measurements were performed at a scan rate of 0.2-0.4 Hz. Three specimens from each group, with three measurements per specimen (n=9/group) were performed. The same three parameters selected for the IFM, also served for data collection and analysis of the AFM.

Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). Morphological examination was performed with scanning electron microscopy (SEM) using a LEO Ultra 55 FEG high resolution SEM (Leo Electron Microscopy Ltd, Cambridge, UK) equipped in combination with an Oxford Inca EDS system (Oxford Instruments Nano Analysis, Bucks, UK) operated between 5 and 10 kV at $\times 5\,000$ to $\times 100,000$ magnification and with high vacuum. The samples were examined after surface sputtering for 60 seconds at 10 mA, resulting in a 30 nm thick gold layer. To describe the atomic composition, EDS analysis at 15 kV and WD 10 at $\times 10,000$ magnification was performed. One specimen from each group was analyzed.

X-ray diffraction (XRD). XRD was performed to analyze the crystalline structure of the samples using a Bruker D8 Advance X-ray diffractometer (Bruker BioSpin Corp., Billerica, MA, USA), collecting a 2θ scanning range of 27-60°, with a step size of 0.05° and with Cu $K_{\alpha 1}$ radiation ($\lambda=1.5405 \text{ \AA}$) as the diffraction light source.

Statistics

The one-way ANOVA, Tukey's test (IBM SPSS Statistics 20, SPSS Inc., Chicago, IL, USA) with the level of significance set to $\alpha = 0.05$ exposed differences in surface structure and flexural strength between the groups.

Systematic review

Study III

Search strategy

Study III sought to obtain an overview of existing methods to achieve bondable surfaces on oxide ceramics, and determine which methods might provide sufficient bond strength, through a systematic review using PubMed (National Center for Biotechnology Information, U.S. National Library of Medicine). The literature search addressed the following questions:

1. What different methods of surface treatments are available to achieve a bond between oxide ceramics and adhesive cement systems?
2. Do any of these methods provide sufficient bond strength for retention of dental restorations without the need for macromechanical retention?

Definitions:

- Oxide ceramics are defined as polycrystalline aluminum oxide and yttrium oxide stabilized tetragonal zirconium dioxide (33).
- A bondable surface is a treated oxide ceramic surface that provides micromechanical interlocking and/or activation for chemical bonding (33).
- Macromechanical retention is defined as the retentive geometric shape of the tooth preparation with the corresponding shape of the dental reconstruction (10).
- Sufficient bond strength is defined by test values ≥ 20 MPa, regardless of the testing methods used (100).

Inclusion and exclusion criteria

To address Questions 1 and 2, the literature review included only the following: original article with abstract included, based on oxide ceramics/polycrystalline oxide ceramics, evaluating the bond strength between oxide ceramics and adhesive cement systems, oxide ceramic bonded to ceramics, composite, enamel or dentine (human or animal), and all test methods for bond strength

performed without the influence of macromechanical retention. For Question 2, inclusion criteria also required: inclusion of a control group, well-defined artificial aging procedure or fatigue simulation artificial aging with TC comprising a minimum of 500 cycles in water with a minimum of 50°C difference between the baths, or long-term storage of 6 months in water at 37°C according to ISO/TS 11405:2003 Dental materials –Testing of adhesion to tooth structure (96), or other test methods with mechanical cyclic loading (>500 cycles) in combination with water/liquid, and verifying the surface of the oxide ceramics before and after testing with more than light microscopy. The exclusion criteria for the addressed questions and aims eliminated: reviews, studies based on glass-infiltrated ceramics or metals or posts, orthodontic brackets made of oxide ceramics, and cement other than adhesive cementation systems (e.g., not resin-modifies glass ionomer cement).

The literature search involved three successive steps (Figure 5). It was conducted from August 2011 to the 1st of January 2012. Free-text words were used as search terms, with English set as the language filter:

(oxide ceramics OR high strength ceramics OR alumina OR aluminum oxide OR zirconia OR zirconium dioxide OR Y-TZP) AND (dental cementation OR dental cements OR adhesive cements OR dental bonding OR resin bond OR resin cements OR luting OR adhesive retention) AND (bond strength)

The literature searches proceeded with *snowballing*, where handsearch of the reference lists of included studies and systematic reviews yields additional relevant articles. Two authors read the titles and further examined any potentially relevant publications and their abstracts, focusing on bond strength analyses between oxide ceramics and adhesive cementation. If at least one author found an article relevant, it was selected for full-text reading using a protocol that indicated inclusions and exclusions criteria (Appendix 1).

Appendix 1. Instructions for *in vitro* studies:

- The alternative “unclear” is used when the data are not possible to obtain from the text.
- The alternative “not applicable” should be selected when the issue is not relevant.
- When clarifying comments are needed, indicate in footnotes

Question	Comments			
	Yes	No	Unclear	Not applicable
<p>1. Study population</p> <p>a) Is it clear how many specimens were included in the study?</p> <p>b) Were any specimens excluded?</p> <p>c) If any specimens were excluded, is it clear how many?</p> <p>d) and why the specimens were excluded?</p> <p>2. Comparability between groups</p> <p>a) Were the groups equal in sample size?</p> <p>b) Were the groups equally treated in an artificial aging procedure/fatigue simulation of the interface (for example, TC)?</p> <p>Supplementary questions</p> <p>a) If TC was done, how many cycles did the specimens undergo and in what temperature range?</p> <p>b) Were the groups stored long-time?</p> <p>c) If long-time storage was done, for how long were the specimens stored?</p> <p>3. Material and method</p> <p>a) Are all subgroups well-defined?</p> <p>b) Were there any control groups?</p> <p>Supplementary questions</p> <p>a) Is the surface treatment commercial?</p> <p>b) Is the surface treatment experimental?</p>				
	Temperature range		Numbers of cycles:	
	Storage time:			
<p>4. Results and precision</p> <p>a) Are the results presented in a way that support and relates to the conclusion?</p> <p>b) What test method was used to measure bond strength?</p> <p>c) How were the surfaces described before and after testing?</p> <p>Supplementary questions</p> <p>a) Is it clear that the significance level was defined in advance?</p> <p>b) Was a power analysis performed?</p> <p>c) Were the results statistically analysed?</p>				
	Test method:			
	Surface analysis:			

RESULTS

In vitro studies

Study I

Bond strength between different bonding systems and densely sintered alumina with sandblasted surfaces or as produced.

Bond strength

The highest bond strengths, which two of the six adhesive cement systems achieved, were significantly higher ($p < 0.001$) compared to the tested adhesive cement systems, irrespective of treatment (artificial aging). There were some differences between the groups with water storage only and those with storage including TC. The Variolink® II group showed a higher bond strength after TC ($p < 0.05$). Table 5a shows shear bond strengths and significant differences between the two subgroups within each group and Table 5b shows them between all the groups (the six adhesive cement systems).

Failure type

Table 6 describes type of failure in the debonded areas in each group. The predominant failure type for both treated (sandblasted) and untreated surfaces were adhesive. All groups, irrespective of pretreatment, showed indications of complex failure.

Table 5a. The results for shear bond strength (MPa): mean values, standard deviation (SD), in the water storage only and thermocycled subgroups of each adhesive cementation system. The statistical difference between the subgroups in each system was determined with Student's t-test.

Groups	Water storage only Mean bond strength (SD)	Thermocycled Mean bond strength (SD)	Statistical significance between the subgroups within the adhesive cement group
Variolink® II	27 MPa (7.0)	34 MPa (5.0)	(p<0.05)
Bisco Chioce™	14 MPa (5.5)	11 MPa (7.6)	(p>0.05)
Bisco Illusion™	10 MPa (3.9)	9 MPa (4.4)	(p>0.05)
Nexus 2™	10 MPa (4.4)	5 MPa (3.6)	(p<0.05)
RelyX™ Veneer	4 MPa (1.7)	2 MPa (1.4)	(p<0.05)
Panavia™ F	31 MPa (4.9)	36 MPa (5.8)	(p>0.05)

Water storage only (storage condition 37°C H₂O), Thermocycled (storage condition 37°C H₂O incl. thermocycling)

Table 5b. Differences in mean shear bond strengths between the water storage only and thermocycled subgroups. The statistical difference between all the subgroups was determined with one-way ANOVA, Tukey's test.

Water storage only	Thermocycled				
	BC	BI	NE	RV	Panavia™ F
Variolink® II	*** ***	*** ***	*** ***	*** ***	NS NS
Bisco Choise™ (BC)		NS NS	NS NS	*** *	*** ***
Bisco Illusion™ (BI)			NS NS	NS NS	*** ***
Nexus 2™ (NE)				NS NS	*** ***
RelyX™ Veneer (RV)					*** ***

Upper /Lower = Water storage only /Thermocycled

NS= Non-statistical significance, ***p≤0.001, **p≤0.01, *p≤0.05

Table 6. Mean percentage of the various failure types in each group according to storage conditions.

Groups	Failure types (Mean percentage) Water storage only				Failure types (Mean percentage) Thermocycled			
	α	β	γ	δ	α	β	γ	δ
Variolink® II	93%	1%	5%	1%	92%	2%	4%	2%
Bisco Choise™	5%	50%	42%	0%	3%	56%	41%	0%
Bisco Illusion™	95%	0%	1%	4%	91%	2%	2%	5%
Nexus 2™	21%	60%	19%	0%	31%	53%	10%	6%
RelyX™ Veneer	74%	4%	20%	2%	44%	3%	50%	3%
Panavia™ F	35%	46%	10%	9%	28%	65%	6%	1%

Water storage only (storage condition 37°C H₂O), Thermocycled (storage condition 37°C H₂O incl. thermocycling)

α = Adhesive failure from the untreated surface, only, β = Adhesive failure from the treated surface, only, γ = Adhesive failure from both the untreated and treated surfaces, δ = Complex cohesive failure type.

Study II

Impactation-modified densely sintered yttria-stabilized tetragonal zirconium dioxide (Y-TZP): Methodology, surface structure and bond strength.

Surface analysis and characterization

Microscopy. The cementation surface of feldspathic porcelain showed a rough surface after etching with HF. The control group (unmodified Y-TZP) showed no visible change in the surface structure. The surface-modified Y-TZP specimens, however, revealed a visibly increased surface structure (Figure 1 a-d).

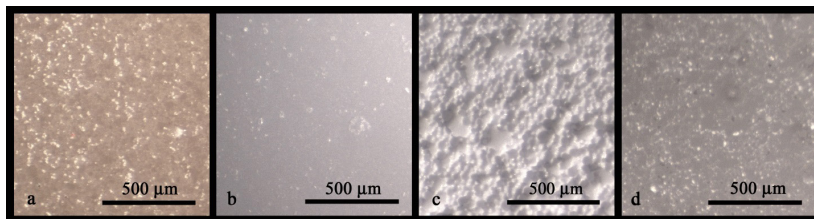


Figure 1 (a-d). Cementation surfaces after etching (x31 magnification). (a): feldspathic porcelain: the glass phase of the cementation surface is removed, leaving a typical surface with micromechanical retention. (b): Y-TZP control: no visible change in surface structure. (c): Y-TZP surface G: the glass residues are removed, leaving a surface with increased surface roughness and enlarged cementation surface (compared to the control group). (d): Y-TZP surface P: an increased surface roughness, and enlarged cementation surface (compared to the control group).

Interferometry. The results from all three parameters (S_a , S_{ds} and S_{dr}) showed a significant difference between the groups that had been surface-modified with glass granulates and all the other groups, both after sintering and after etching, Table 7. Figure 2 (a-f) shows images of the various surfaces, including unmodified surfaces and those modified with glass or polymer.

Table 7. Mean values and standard deviation (SD) of tridimensional roughness parameters (S_a , S_{ds} and S_{dr}) as determined by IFM (scanning area of $200 \times 260 \mu\text{m}^2$).

Surface treatment	S_a (μm)	(SD)	S_{ds} (μm^2)	(SD)	S_{dr} (%)	(SD)
	Mean		Mean		Mean	
Control sintered	0.15 ^a	(0.02)	174611 ^a	(8555)	2.85 ^a	(0.34)
Control etched	0.10 ^b	(0.01)	143196 ^{a,b}	(9286)	0.92 ^b	(0.23)
Surface G sintered	5.03 ^{a,c,e}	(0.48)	293188 ^{a,b,c}	(8694)	754 ^{e,d}	(114.5)
Surface G etched	4.07 ^{a,b,d,e}	(0.57)	249765 ^{a,b,d}	(6822)	142 ^{e,d}	(40.9)
Surface P sintered	0.26 ^{c,d}	(0.11)	158313 ^{a,c,e}	(29185)	11.9 ^c	(12.8)
Surface P etched	0.28 ^e	(0.33)	179152 ^{a,b,d,e}	(7949)	7.62 ^d	(15.09)

^{a, b, c} Groups labeled with the same superscripted letter indicate significant statistical difference in surface roughness ($p \leq 0.05$) between the groups.

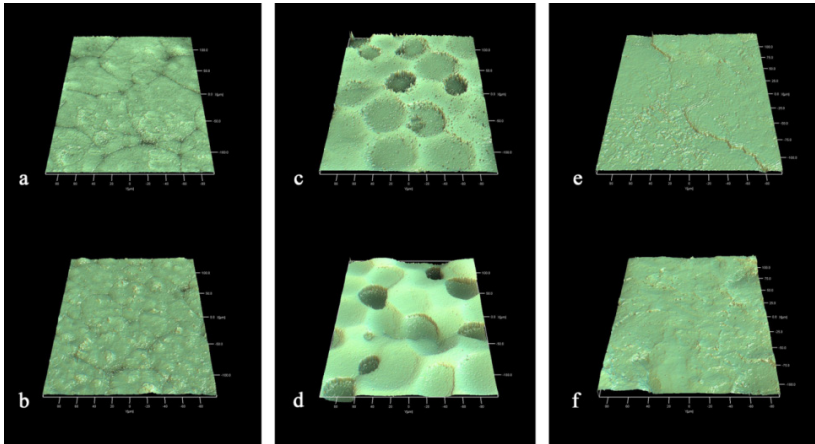


Figure 2 (a-f). Cementation surfaces after sintering a,c,e and after etching b,d,f: (a): Y-TZP control: clearly defined grain boundaries. (b): Y-TZP control: small changes in surface structure can be observed after etching. (c): Y-TZP surface G: creates a rough surface structure. (d): Y-TZP surface G: the glass residues are removed leaving a surface with increased surface roughness (compared to the control group) but not as rough as before etching the same surface. (e): Y-TZP surface P: creates a rough surface structure. (f): Y-TZP surface P: the surface structure is rougher compared to the same non-etched surface, but is not statistically significant.

Bond strength

All groups with modified cementation surfaces showed significantly higher bond strength compared to their corresponding control group. The glass-modified surface in combination with the Variolink® II cement system, showed the highest bond strength. Table 8 lists the results.

Failure type

The surface-modified groups showed mainly cohesive failure (24 of 32) in which the failure occurred in the feldspathic porcelain, while all failures in the two control groups were adhesive. There were no significant differences between groups with the same surfaces, irrespective of cement system, Table 8.

Table 8. The results for bond strength (MPa): mean values, standard deviation (SD), maximum and minimum values and failure types between the groups, the number (n) of adhesive and cohesive failures and statistical differences.

Groups	Abbreviation	Bond strength		Maximum/Minimum value	Failure type Adhesive/Cohesive ⁽ⁿ⁾	
		Mean value	SD			
Variolink® II + surface G	VA-G	34.9 ^a	(5.8)	42.4	25.8	2 / 6
Variolink® II + surface P	VA-P	30.9 ^b	(7.7)	40.2	20.6	0 / 8 ^a
Variolink® II + control	VA-C	20.5 ^{oc}	(4.2)	27.8	15.5	8 / 0 ^a
Panavia™ F + surface G	PA-G	26.1 ^{od}	(4.0)	31.8	21.6	2 / 6
Panavia™ F + surface P	PA-P	29.6 ^{oc,e}	(3.8)	35.8	24.7	4 / 4
Panavia™ F + control	PA-C	17.8 ^{ob,d,e}	(3.5)	25.1	13.1	8 / 0 ^a

Adhesive denotes debonding between the cement and the Y-TZP or porcelain. Cohesive denotes fracture in the feldspathic porcelain.

^{a, b, c} Groups labeled with the same superscripted letter indicate significant statistical difference in bond strength and fracture type ($p \geq 0.05$) between the groups.

Study IV

Surface structure and mechanical properties of impaction-modified Y-TZP.

Topographical surface characterization by IFM and AFM

The surface structure of sandblasted Y-TZP differed from glass-modified Y-TZP. In both resolutions investigated, micrometer and nanometer, the glass-modified surface was rougher. Table 9 shows the parameters calculated from the IFM and AFM and Figure 3 (a-h) shows images of the surface structures. The results of the IFM, indicate a significant difference ($p < 0.05$) between both the unmodified Y-TZP (CE, CS, CSE) and sandblasted with or without the etching procedure (CSS, CSSE), and the glass-modified groups, which showed a rougher surface. Among the glass-modified groups, those that were sintered (GS) or sintered and etched (GSE) had the roughest surfaces.

Results from the AFM found no significant differences between the groups for the parameter S_a . The S_{ds} parameter showed a significant difference between the group that were etched before sintering (CE), as well as the unmodified surface with or without the etching procedure (CSE, CS), and the groups that were sandblasted and etched (CSSE) or glass-modified (GS, GSE). However, there was no significant difference found between the sandblasted and glass-modified groups. The S_{dr} parameter showed significantly higher values for the group with the sandblasted surface than for the groups that were etched before sintering (CE) or just sintered (CS).

Table 9. Mean values and standard deviation (SD) of tridimensional roughness parameters (S_a , S_{db} and S_{dr}) as determined by IFM (scanning area of $200 \times 260 \mu\text{m}$) and AFM (scanning area of $10 \times 10 \mu\text{m}$).

Groups	IFM		AFM			
	S_a (μm) Mean (SD)	S_{db} (μm^2) Mean (SD)	S_{dr} (%) Mean (SD)	S_o (nm) Mean (SD)	S_{db} (μm^2) Mean (SD)	S_{dr} (%) Mean (SD)
CE	0.24 (0.21) ^b	178971 (14550) ^o	8.3 (1.9) ^o	37.0 (5.4)	6.6 (0.98) ^o	7.4 (1.6) ^o
CS	0.27 (0.02) ^b	161326 (5761) ^b	8.7 (0.98) ^b	35.3 (3.5)	7.5 (1.6) ^b	6.9 (1.2) ^b
CSE	0.29 (0.29) ^c	140941 (6902) ^{oc}	11.0 (1.9) ^c	41.5 (16.8)	7.6 (3.4) ^c	9.0 (5.5)
CSS	0.47 (0.02) ^d	191903 (1613) ^{bd}	15.8 (0.44) ^d	58.4 (12.8)	15.5 (5.7)	27.3 (14.9) ^{o,b}
CSSE	0.50 (0.04) ^e	170396 (4733) ^{ce}	24.6 (2.0) ^e	51.1 (12.4)	22.5 (6.2) ^{oc}	14.5 (9.1)
GE	4.2 (0.70) ^{oe}	227139 (19254) ^{of}	207 (65) ^{of}	41.5 (10.3)	13.5 (5.1) ^d	11.6 (5.8)
GS	4.3 (0.29) ^{of}	278845 (18650) ^{of}	368 (13.4) ^{of}	40.2 (13.8)	23.8 (11.6) ^{od}	15.8 (15.2)
GSE	3.8 (0.57) ^{of}	297974 (21567) ^{of}	322 (90) ^{of}	62.2 (50.1)	21.0 (7.6) ^{oc}	22.6 (27.5)

CE: Control; etched before sintering, CS: Control; sintered, CSE: Control; sintered, etched, CSS: Control; sintered, sandblasted, CSSE: Control; sintered, sandblasted, etched, GE: Glass-modified; etched before sintering, GS: Glass-modified; sintered, GSE: Glass-modified; sintered, etched.

^{a, b, c} Groups labeled with the same superscripted letter indicate significant statistical difference in surface roughness ($p \geq 0.05$) between the groups.

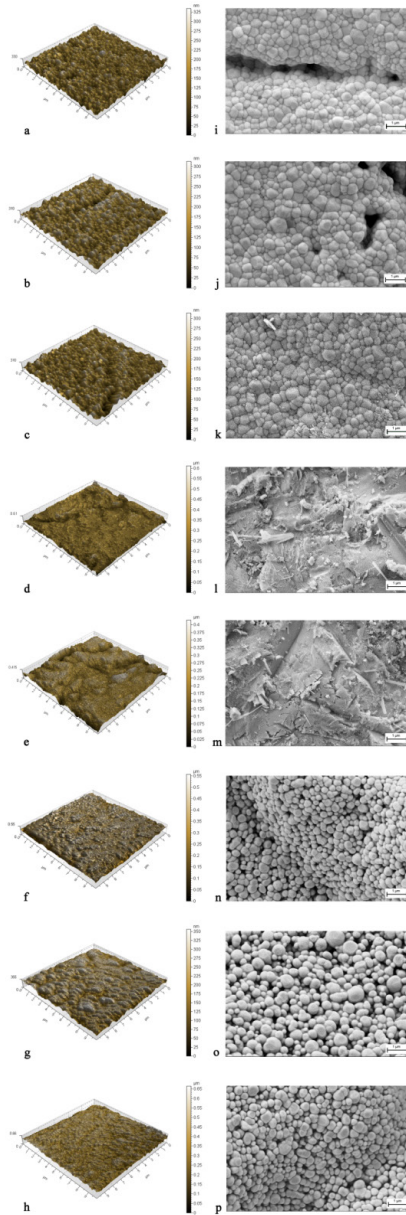


Figure 3 (a-p). AFM images ($10 \times 10 \mu\text{m}$) and SEM electromicrographs ($\times 30,000$ magnification) of each group: CE: Control; etched before sintering (a and i), CS: Control; sintered (b and j), CSE: Control; sintered, etched (c and k), CSS: Control; sintered, sandblasted (d and l), CSSE: Control; sintered, sandblasted, etched (e and m), GE: Glass-modified; etched before sintering (f and n), GS: Glass-modified; sintered (g and o) and GSE: Glass-modified; sintered, etched (h and p).

Surface morphology and chemical surface characterization by SEM and EDS

Figure 3 (i-p) depicts SEM images of surface morphology. The chemical composition of glass-modified Y-TZP differed from unmodified Y-TZP. A phase transformation was identified in the glass-modified Y-TZP. While the EDS analysis found oxygen (O), zirconium (Zr), yttrium (Y) or hafnium (Hf), carbon (C), boron (B), and aluminum (Al) on the surface of both the unmodified and sandblasted Y-TZP, the components on the glass-modified Y-TZP surface were O, Zr, C, B and silicon (Si), sodium (Na), calcium (Ca), and fluorine (F). Figure 4a summarizes the chemical compounds identified.

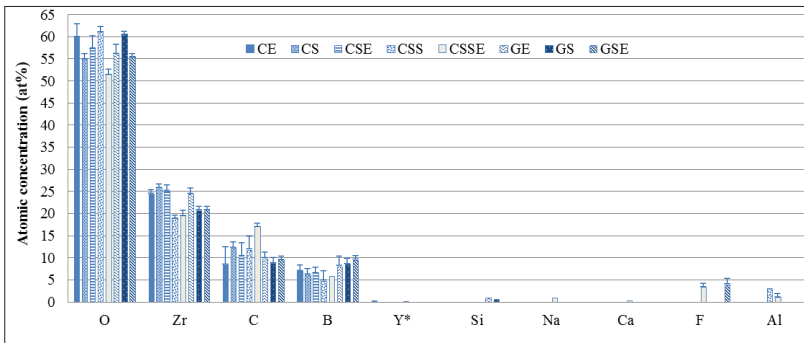


Figure 4a. Mean values and standard deviation (SD) of atomic concentration (at %) of elements according to the sample groups, as determined by EDS analysis.

*Yttria was identified primarily, although it could also be hafnium which has been suggested might be present.

Identification of crystalline structure by XRD

The XRD results showed that two crystalline structures dominate, the monoclinic phase and the tetragonal phase, Figure 4b. Unmodified Y-TZP samples showed only the tetragonal phase. After sandblasting the Y-TZP, monoclinic phase was identified too. The glass-modified Y-TZP showed increase monoclinic structure and less of the tetragonal phase. Etching procedures showed no differences, regardless of group. Thus etching will not affect the crystalline structure.

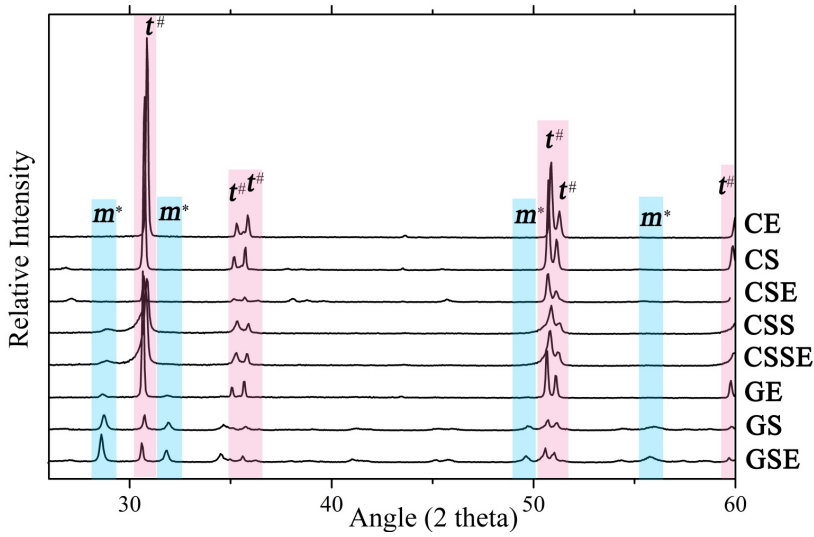


Figure 4b. XRD diffractograms of samples from each group.

*m: peak indicating the monoclinic phase, #t: peak indicating the tetragonal phase. CE: Control; etched before sintering, CS: Control; sintered, CSE: Control; sintered, etched, CSS: Control; sintered, sandblasted, CSSE: Control; sintered, sandblasted, etched, GE: Glass-modified; etched before sintering, GS: Glass-modified; sintered and GSE: Glass-modified; sintered, etched.

Biaxial flexural strength

The groups with no glass modification showed significantly higher flexural strength compared to the glass-modified groups ($p < 0.001$). They also showed increased flexural strength after sandblasting ($p < 0.001$). Adding cement to the sandblasted surface increased flexural strength even more ($p < 0.01$). After TC, however, the cement layer on both the unmodified and the sandblasted surfaces had air pockets, and part of the cement was loose. This was not seen in the glass-modified groups. Table 10 summarized the results.

Table 10. The results for flexural strength (MPa): mean values, standard deviation (SD), maximum and minimum values and statistical differences.

Groups	Abbreviation	Mean (SD)	Maximum	Minimum
Control, sintered	CS	443 (64) ^a	549	252
Control, sintered + cement	CSC	476 (63) ^b	600	327
Control, sintered + sandblasted	CSS	574 (53) ^{a,c}	686	488
Control, sintered + sandblasted + cement	CSSC	624 (68) ^{a,d}	782	492
Glass-modified, etched before sintering	GE	237 (49) ^{a,e}	420	160
Glass-modified, etched before sintering + cement	GEC	250 (37) ^{a,d,f}	325	192
Glass-modified, sintered + etched	GSE	187 (20) ^{a,g}	222	147
Glass-modified, sintered + etched + cement	GSEC	235 (33) ^{a,d,g}	346	193

^{a, b, c} Groups labeled with the same superscripted letter indicate significant statistical difference in fracture strength ($p \geq 0.05$) between the groups.

Systematic review

Study III

Bonding between oxide-based ceramics and adhesive cement systems: A systematic review.

Literature identification

Figure 5 is a flow diagram of the number of retrieved, potentially relevant publications and the publications included and excluded in each step of the data extraction. The total number of publications included was 127 publications, of which 23 examined the question “Do any of the methods provide sufficient bond strength for the retention of dental restorations without the need for macromechanical retention?”.

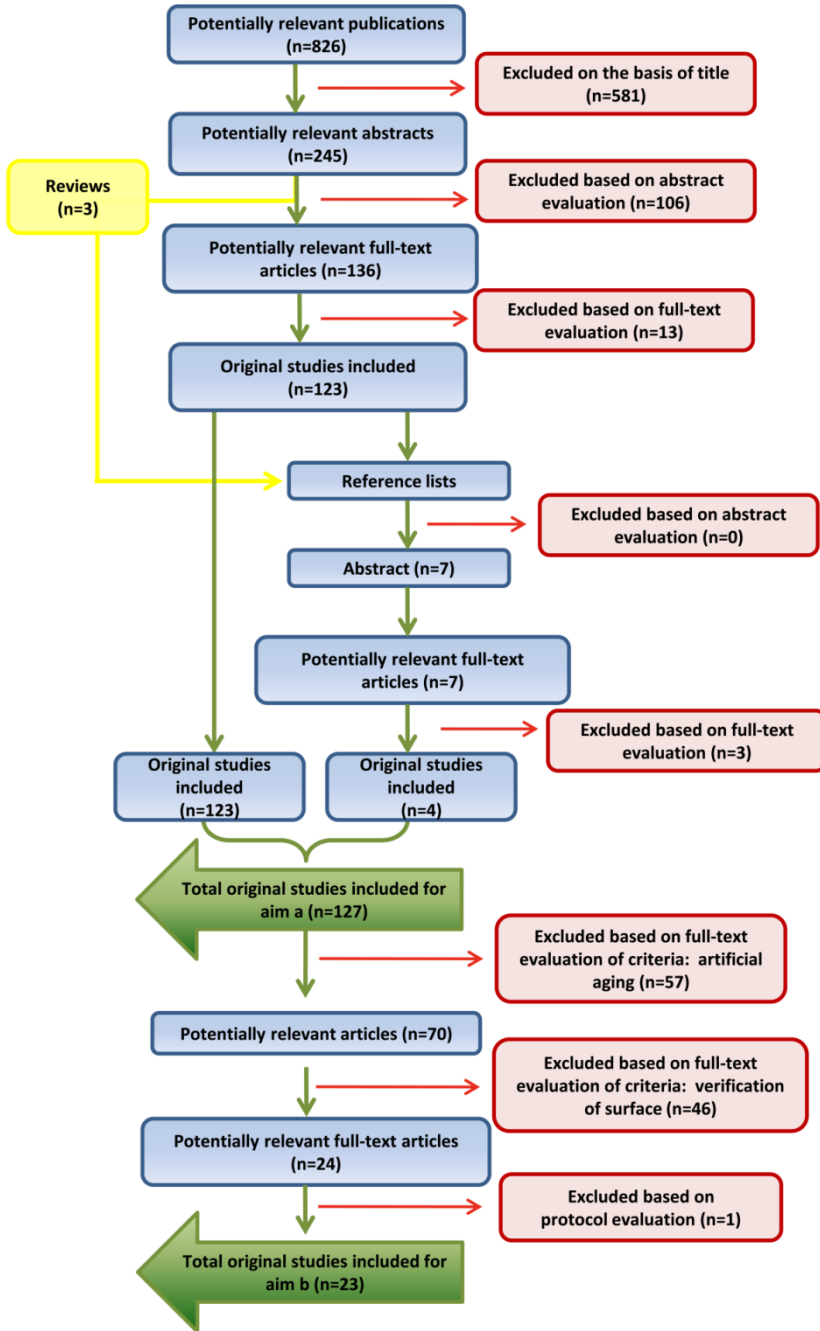


Figure 5. Search strategy and results of the literature review

Interpretation of data on surface treatment of oxide ceramics and bond strength

Figure 6 presents the various methods of surface treatment that aims to create a bondable cementation surface in oxide ceramics. The surface treatments are divided into seven main categories: as-produced, grinding/polishing, airborne particle abrasion, surface coating, laser treatment, acid treatment, and primer treatment. Each group has own specific surface treatments, followed by a variety of cleaning procedures with water, ethanol, acetone or phosphoric acid and then various cement systems. The evaluation did not consider cleaning and cementation procedures.

Oxide ceramics

Concerning Question 2, the 23 studies included 12 different brands for stabilized zirconium dioxide (28, 61, 72, 76, 78, 88, 89, 101-114) and two of aluminum oxide (61, 89, 94, 113).

Artificial aging

Artificial aging procedure differed in the various studies. For TC, the number of cycles ranged from 500 to 100,000 at water temperatures of 5°C to 60°C (72, 78, 88, 89, 101, 102, 104-107, 110-112, 115) or TC (20,000 or 37,500 cycles) with long-term water storage (37°C) for 90 or 150 days (28, 61, 76, 94, 108, 109). Other artificial aging procedures included long-term water storage, varying from 24 weeks to 104 weeks, (103, 104, 110, 113) and cyclic impaction and compressive load in water with up to 1,000,000 cycles (114).

Test method

The most common test method (16 of 23) to evaluate bond strength was the shear bond strength test (SBS/ μ SBS) (61, 72, 76, 78, 88, 89, 101, 102, 105-107, 111-115) (Table 11).

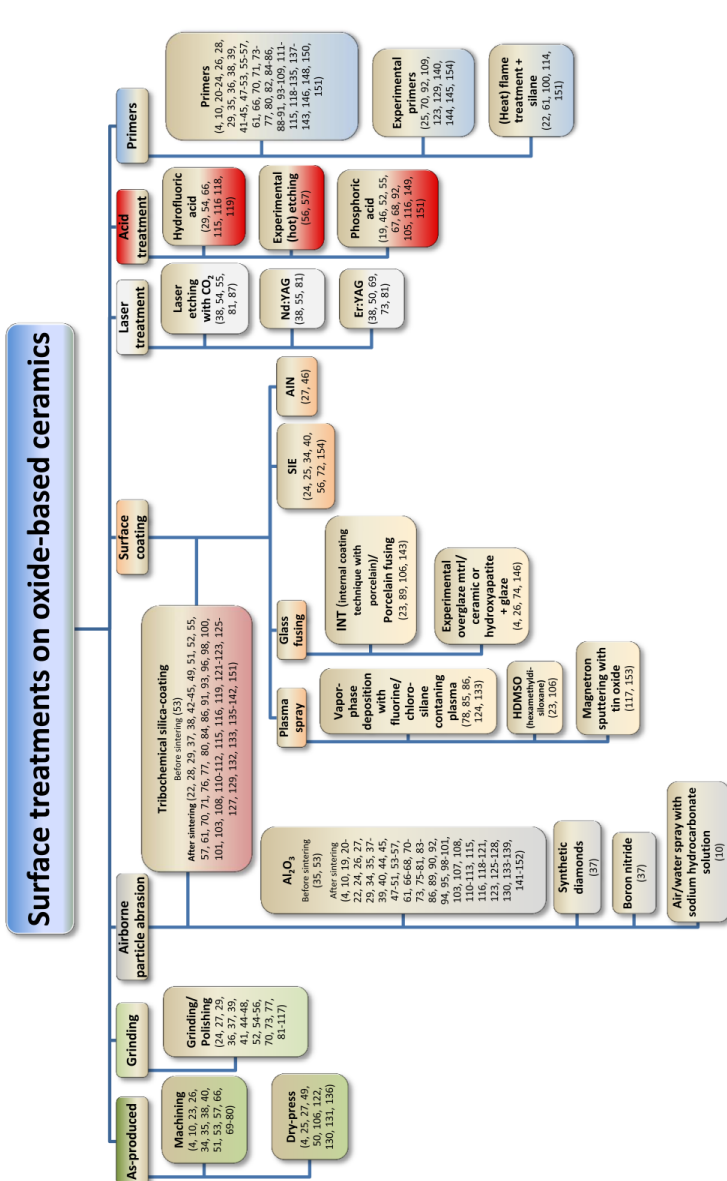


Figure 6. A schematic illustration and overview of the various surface treatments from the literature review. The reference number is in accordance with the original published study (see Study III in the appendix).

Table 11. The results from the included studies (n=23). Ceramic material, surface treatment, cement system, type of artificial aging and test methods.

Article	Materials	Cementation systems	Artificial aging TC/ LT storage	Test method
Phark JH, et al. 2009 (61)	Procera Alumina, Nobel Biocare Procera Zirconia, Nobel Biocare bonded to composite resin	Clearfil Esihetic + Clearfil Ceramic primer RelyX ARC + RelyX Ceramic primer	90 days of water storage including 20,000 cycles (5°- 60° C)	SBS
Phark JH, et al. 2009 (76)	Procera Zirconia, Nobel Biocare bonded to composite resin	Panavia F2.0 RelyX ARC RelyX Unicem	90 days of water storage (RT) including 20,000 cycles (5°- 60° C)	SBS
Jevnikar P, et al. 2010 (78)	TZ-3YB-E zirconia powder, Tosoh bonded to composite resin	RelyX Unicem	12,000 cycles (5°-55° C) after 24 hours of water storage (37° C)	SBS
Matinlinna JP, et al. 2006 (88)	Procera AllZircon, Nobel Biocare bonded to cements	Experimental Bis-GMA, Röhm RelyX ARC	6 000 cycles (5°-55° C) after 24 hours of storage in a desiccator in RT.	SBS
Abousheib MN. 2011 (110)	Procera Zirconia, Nobel Biocare bonded to composite resin	PanaviaF 2.0	10,000 cycles (5°-55° C) or 4-26- 52-104 weeks in water (37° C)	µTBS
Moon JE, et al. 2011 (111)	ZrO ₂ Rainow, Denitum bonded to composite resin	Clearfil SA luting cement Zirconite RelyX Unicem Superbond C&B Multilink	5 000 cycles (5°-55° C) after 24 hours of water storage (37° C)	SBS

(Table 11 continues on next page)

Article	Materials	Cementation systems	Artificial aging TC/ LT storage
Koizumi H, et al. 2010 (115)	Alumina, Furuuchi Chemical Corp. bonded to cement with 8 different primers	Unprimed as control Eight primers: 1) Alloy Primer 2) Eye Sight Opaque primer 3) Estenia Opaque Primer 4) MR. Bond 5) M.L Primer 6) Super-Bond Liquid 7) All Bond II Primer B 8) Acryl Bond Luting agent: Super-Bond Cat. + Liquid + Opaque powder	100,000 cycles (5°-55° C) after 24 hours of water storage
Kulunk S, et al 2011 (112)	ICE Zircon translucent, Zirconzahn bonded to composite	PanaviaF 2.0	6 000 cycles (5°-55° C) after 24 hours of water storage (37° C)
Akyil MS, et al. 2010 (72)	Zirconia, Copran Zirconia blank, White Peaks Dental system bonded to composite	Clearfil Esthetic Cement	5 000 cycles (5°-55° C) after 24 hours of water storage (37° C)
Yun JY, et al. 2010 (107)	ZrO ₂ , Rainow, Dentium bonded to cements	Alloy primer+ PanaviaF2.0 V-primer + Superbond C&B Metalite + M bond	5 000 cycles (5°-55° C) after 24 hours of water storage (37° C)
Aboushelib MN, et al. 2010 (104)	LAVA Zirconia, 3M ESPE Dental Products bonded to composite	PanaviaF 2.0	10,000 cycles (5°-55° C) or 4 weeks in water (37° C) or 20 weeks in water (37° C)

(Table 11 continues on next page)

(Table 11 cont.)

Nakayama D, et al. 2010 (105)	KATANA™ Zirconia, Noritake bonded to cement	Unprimed as control Eight primers: 1) Alloy Primer 2) Eye Sight Opaque primer 3) Estenia Opaque Primer 4) MR. Bond 5) M.L. Primer 6) Super-Bond Liquid 7) All Bond II Primer B 8) Acryl Bond Luting agent: Super-Bond Cat. + Liquid + Opaque powder	10,000 cycles (5°-55°C) after 24 hours of water storage (37°C)	SBS
Heikkinen TT, et al. 2007 (89)	LAVA Zirconia, 3M ESPE Dental Products. Procera Alumina or Procera Zirconia, Nobel Biocare bonded to composite	3M Multipurpose resin	6 000 cycles (5°-55°C)	SBS
Matinlinna JP, et al. 2007 (102)	Procera AllZircon, Nobel Biocare bonded to cement	RelyX ARC	6 000 cycles (5°-55°C)	SBS
Kumbuleglo O, et al. 2006 (101)	DCS Zirconia bonded to cement	Panavia F RelyX Unicem	2 000 cycles (5°-55°C) after 24 hours of water storage (37°C)	SBS
Takeuchi K, et al. 2010 (106)	KATANA™ Zirconia, Noritake bonded to Ti, JIS grade 2	RelyX ARC	30,000 cycles (5°-60°C) after 24 hours of water storage (37°C)	SBS
Zhang S, et al. 2010 (109)	ZrO ₂ Cercon bonded to composite resin	PanaviaF 2.0	150 days of water storage (37°C) including 37,500 cycles (5°-55°C)	TBS

(Table 11 continues on next page)

(Table 11 cont.)

Article	Materials	Cementation systems	Artificial aging TC/ LT storage
Yang B, et al 2010 (108)	Zirconia, Cercon ceramic bonded to cement	Multilink (automix)	150 days of water storage (37°C) including 37,500 cycles (5°-55°C)
Kern M, et al 2009 (28)	Zirconia, Cercon ceramic bonded to cement	Multilink (automix)	150 days of water storage (37°C) including 37,500 cycles (5°-55°C)
Hummel M, Kern M. 2004 (94)	Procera Alumina, Nobel Biocare bonded to Clearfil F2	Variolink II + Heliobond, = System Variolink II system Alloy primer + Variolink system Monobond-S + Variolink II system Superbond C&B Panavia 21	150 days of water storage (37°C) including 37,500 cycles (5°-55°C)
Foxton RM, et al. 2011 (113)	Procera Alumina or Procera Zirconia, Nobel Biocare bonded to cements	NAC-100 Variolink II	6 months of water storage (37°C)
Oyague RC, et al. 2009 (103)	Zirconia Cercon bonded to composite resin	Clearfil Esthetic Cement RelyX Unicem Calibra	6 months of water storage (37°C)
Kawai N, et al. 2011 (114)	YPSZ, Nikkato bonded to each other	SuperBond C&B Panavia Fluora Cement Fuji Luting (RRGC)	Cyclic impaction Compressive cyclic load or Shear cyclic impaction load numbers of cycles 1-10 ⁶

Table 12. Mean values from tests of various surface treatments.

Surface treatment	Included studies		Test methods (range of mean values, MPa)			No. of studies with mean values >20 MPa	
	Question 1 and 2		TBS	μTBS	SBS		
As-produced	Q1 (34/127)		0	0-8	0-17	11-20	No study
	Q2 (10/23)						
Grinding/polishing	Q1 (56/127)		0	*	0-36	*	1 study ⁽¹¹⁵⁾
	Q2 (8/23)						
Airborne particle abrasion	Q1 (94/127)		0-30	0-27	0-31	9-26	9 studies ^(28,72,94,104,108,110,113)
	Q2 (15/23)						
Tribochemical silica-coating	Q1 (55/127)		25	0-15	0-45	*	7 studies ^(72,89,94,101,106,112,114)
	Q2 (10/23)						
NobelBond	Q1 (8/127)		*	*	1-41	*	1 study ⁽⁶¹⁾
	Q2 (2/23)						
SIE	Q1 (7/127)		*	43-53	*	*	2 studies ^(104,110)
	Q2 (2/23)						
AlN	Q1 (2/127)		9-33	*	24-28	*	2 studies ^(78,109)
	Q2 (2/23)						
Laser etching	Q1 (8/127)		*	*	15-22	8-17	1 study ⁽⁷²⁾
	Q2 (2/23)						
Primer treatment	Q1 (98/127)		0-38	*	0-45	*	5 studies ^(28,94,106,108,115)
	Q2 (9/23)						

*Values not available from the specific test method

SIE: Selective infiltration etching, AlN: Coating with nanostructured alumina powder.

Definition of the different surface treatments

Table 12 shows the mean test values for the surface treatments described below.

As-produced: Studies that have kept the cementation surface as-produced, some milled from pre-sintered or fully sintered blocks and other pressed, according to the production process of the respective manufacturer and material.

Grinding/polishing: Studies examining ground and/or polished surfaces obtained using diamond wheels and/or various grit silicon carbide paper (grit size range: 120-1,500 grit).

Airborne particle abrasion (sandblasting): This can be performed using aluminum oxide, diamond, or boron nitride particles, with particle sizes ranging from 25 to 250 μm , and various air pressures and distances during blasting. It is one of the most common mechanical surface roughening treatments.

Surface coating: The literature described a variety of coating processes. Tribochemical silica-coating is a surface treatment that forms a silica layer by means of airborne particle abrasion using special silica-coated aluminum oxide particles. It is used either as a combined surface treatment, for mechanical roughening and surface coating. Tribochemical coating was one of the most widely used surface treatments.

Other surface treatments included plasma spraying, glass fusing, selective infiltration etching (SIE), and coating with nanostructured alumina powder (AlN). Plasma spraying can be carried out using various coating techniques, such as chemical or physical vapor deposition, to apply plasma coatings, for example fluorine, chlorosilane, hexamethyldisiloxane, or tin oxide.

Glass fusing is an internal coating technique which can be carried out using a number of different materials, including commercial porcelain, ceramics or experimental overglaze material, before being finished with porcelain fusing.

The infiltration technique (SIE) is a coating technique followed by an etching procedure. A thin layer of glass conditioning agent is sprayed onto the cementation surface of Y-TZP and then heated. The molten glass diffuses at the grain boundary regions. Excess glass is subsequently removed with HF prior to the cementation.

AlN technique, in brief, involves coating the restoration with nanostructured alumina powder (nitride of aluminum) by immersing it in a solution in which the dispersed AlN powder begins to decompose, forming a nanostructured boehmite coating on the surface.

Laser treatment: Laser etching can be done with carbon dioxide (CO₂), neodymium-doped yttrium aluminum garnet (Nd:YAG) or erbium-doped yttrium aluminum garnet (Er:YAG). The surface of the oxide ceramic is irradiated with lasers to roughen it in order to create micromechanical interlocking.

Acid treatment: HF or phosphoric acid treatment prior to cementation is the most common acid treatment. It is included in the cementation procedure as a cleaning method and is not always specified as a surface modification per se. Some studies evaluated an experimental etching with heat or chemical etching with alkaline hydroxylation.

Primer treatments: The majority of the reviewed studies used an adhesive cementation system that included a primer. Some studies evaluated uses of primer, including commercially available primers and experimental primers, in combination with heat or flame treatment.

DISCUSSION

Three *in vitro* studies and a systematic review examined both general and specific knowledge regarding surface treatment of oxide ceramics, as well as the bond strength between them and adhesive cement systems.

Methods: In vitro studies

In vitro studies are often used to evaluate materials or designs of restorations and are relatively efficient in terms of time and cost compared to *in vivo* studies. The purpose of *in vitro* studies is to be able to test dental materials and specific factors in a controlled laboratory environment, with methods that are standardized and mimic clinical situations as closely as possible (116). The goal of laboratory testing the materials is to be able to correctly predict clinical performance before materials are tested and evaluated in a clinical situation. *In vitro* studies have several limitations, however, arising from the fact that material testing in a laboratory is simplified and does not fully capture the complexity of the physical and mechanical properties of substructures, the design of the restoration, the biological environment of the oral cavity, or the loads of chewing. This makes direct comparison of laboratory results to clinical situations difficult. Rather, *in vitro* studies should be seen as a step in predicting clinical performance (57, 117, 118).

The choice of included materials

Oxide ceramics - aluminum oxide and yttrium oxide stabilized tetragonal zirconium dioxide

The clinical applications of oxide ceramics have increased in comparison to PFM. Due to material and process developments of oxide ceramics, aluminum oxide-based restorations have been replaced by stabilized zirconium dioxide-based restorations. Looking ahead, there will likely be further development of translucent Y-TZP and its dental applications, for instance in multiple FDP units (35) and RBCBs (53).

The blocks and the cylinders that were the specimens in Study I were all made of densely sintered aluminum oxide. This choice was made to simplify the study design. By having the same oxide ceramic material, both ceramic surfaces could be tested equally (119). The literature shows that it is more common to use different materials, such as composite cylinders bonded to oxide-based ceramic substrate (58, 61, 76, 78, 87, 89, 94, 103, 104, 109, 111, 112). One advantage of having composite is that the bond in the interface between adhesive cement system and composite is well established. Another benefit is that the composite is easy to handle and specimens can be produced easily. One of the drawbacks is that each material has a different elastic modulus, which influences the interfaces and the received load, and affects the bond strength and the type of failure. Bond strength tends to increase with the use of materials with high elastic modulus (119, 120).

Study II presents a novel method for creating a bondable cementation surface on Y-TZP. The method uses modified-additive production in conjunction with two different impaction mediums. The use of the glass medium was based on the knowledge that a bondable surface is created by etching glass-containing ceramics (24, 25). Using HF on feldspathic porcelain and glass ceramics creates a rough, mainly crystalline surface with pits and microlacunae. The surface glass is almost completely removed but the crystal phases are not markedly affected by the acid and hence remain substantially unchanged after etching. The etched surface enhances retention by interlocking with the cement, thus creating micromechanical retention while the small portion of glass residues on the surface enhances the chemical bond between the cement and the ceramic

surface (24, 48). The glass granules themselves can be etched and the glass-modified Y-TZP surface can be treated as a glass-containing ceramic. To evaluate if micromechanical retention was sufficient or if chemical retention was needed as well, polymer granules were used as another medium. The specimens consisted of blocks made of feldspathic porcelain and cylinders made of Y-TZP. The choice of feldspathic porcelain was based on its large proportion of glass, which can be etched to create better conditions for micromechanical retention and thereby a strong adhesive bond (24). The feldspathic porcelain was intended to act as a reference for the tested surfaces of Y-TZP. Where the feldspathic porcelain fractured, however, there were cohesive failures. Thus, the achieved bond strength was higher than the cohesive strength of the porcelain. Both parts of the specimen could have been made of the same material, as in Study I.

Study IV followed up and further developed the results from Study II, evaluating surface-modified Y-TZP, in particular the glass-modified Y-TZP. The study investigated the influence of the glass granules and the effect of etching the cementation surface on chemical composition and strength of the surface-modified Y-TZP. Using glass granules embedded prior to sintering meant the glass would melt but still be present after sintering. This glassy surface must be removed before cementation to allow micromechanical retention. The etching with HF may leave small amounts of glass residues in the surface structure where the etch fails to act. This residues promotes chemical retention between the glass-containing Y-TZP surface and the primer, and thus further increases bond strength beyond the micromechanical retention created (24, 48, 93). The glass residues may also influence the physical properties of the Y-TZP material, especially if phase transformation of the material's crystals occurs (31, 40, 42). The etching procedure was done both before and after the sintering of the untreated and glass-modified Y-TZP. With etching before the sintering of the glass-modified Y-TZP, the sintering process could be more beneficial because of a more homogeneous sintering of the ceramic powder. It resulted in a dense material for which no regard to the difference in the coefficient of thermal expansion (TEC) between the glass medium and the Y-TZP needed to be taken. The difference in the TEC of the materials might influence the properties of the final restorations by creating inherent stresses and weaken its mechanical properties (29).

Adhesive cement systems

Study I investigated six adhesive cement systems which, at the time of the study, were recommended by various cement manufactures for adhesive bonding to oxide ceramics. Adhesive cement systems vary in composition (one-paste or two paste systems), curing (chemical, light-cured, or dual-cured), type and amount of fillers (nano, micro, and hybrid) and viscosity (low, medium, or high). Other differences include the physical and chemical properties of the cement, which in turn depend upon degree of conversion in the cement (how many of the reactive sites on the polymer chains were activated as the cement set) (121). Five of the chosen adhesive cement systems were dual-cured, of which four were Bis-GMA-based and one, Panavia™ F, was a MDP-containing resin cement. The sixth, RelyX™ Veneer Cement, differed from the other cement systems in that it was light-cured (Table 3).

Study II tested the two cements, Variolink® II and Panavia F™2.0, that showed superior results in Study I. Both cements are well documented and have shown stable bond strength to oxide ceramics compared to other adhesive cements (52, 55, 94). Variolink® II is considered to be primarily for use with feldspathic porcelain or glass ceramics (122). The blocks of feldspathic porcelain combined with the use of Variolink® II could serve as reference for the surfaces to be tested. The hypothesis that the surface of glass-modified Y-TZP contains glass also suggested choosing Variolink® II. The phosphate monomer-containing Panavia F™2.0 system, primarily developed for metal restorations, has shown that it bonds chemically to metal oxides and oxide-based ceramics (5, 55, 56, 64).

The choice of cement systems in Study IV was based on previous studies, which showed that the MDP-containing cement Panavia F™2.0 bonds to oxide-based ceramics (55, 56, 64). This cement was used partly to investigate whether an adhesive bond could be achieved on an unmodified surface compared to a modified surface, and partly to seal the surfaces, in particular the glass-modified surface, in order to reinforce the material (7, 101). Since LTD starts at the surface, surface cementation might slow the aging process and maintain the mechanical properties of the oxide ceramic (29).

The choice of processing and surface treatment

Processing technology

The specimens in Study I were industrially manufactured by CIP technology under controlled conditions with the optimal material properties for a dental restoration. In Studies II and IV, the specimens were manufactured by uniaxial pressure in a modified additive production, since the production was a methodological development, with different mediums embedded into the surface of Y-TZP, aiming to create a bondable surface of oxide ceramics. During uniaxial pressing, the distribution of pressure on the ceramic powder might have been uneven, especially against the walls of the mold, where areas of low particle packing result in an anisotropic material (50, 51). This could have been avoided with isostatic pressing, in which the pressure is applied from all directions, resulting in a more homogenous and dense material. Probably, the mechanical properties would have been better and the biaxial flexural strength higher (123).

Pretreatment of the cementation surface and cleaning process

In Study I, the cleaning process and surface treatment of the cementation surface of the blocks involved first cleaning with acetone, then sandblasting with 110 μ m Al₂O₃ particles, and finally ultrasonic cleaning in 96% isopropanol. Only the cylinders were subjected to the cleaning process. This was done for all specimens; for specimens in the groups with the Panavia™ F system, the cleaning process also included phosphoric acid treatment. In Studies II and IV, all cementation surfaces of the specimens (including blocks, cylinders and discs) were etched with HF. This replaced previous cleaning process from Study I, partly because it is the standard procedure for etching feldspathic porcelain and partly because HF has been shown to be effective in cleaning the cementation surface of ceramic restorations. Choosing HF cleaning also allowed all cementation surfaces to be etched and cleaned in the same way, whether feldspathic porcelain, glass-modified Y-TZP, or Y-TZP. In Study II, after etching with HF, the cementation surfaces were cleaned with phosphoric acid before cementation. The reason for this cleaning was to mimic the clinical situations, where restorations need to be cleaned of contaminations after try-in in the mouth

(124-127). At the time of testing, using phosphoric acid to clean the cementation surface was the clinical procedure recommended by the manufacturer. Phosphoric acid cleaning was excluded from Study IV, however, because of its potential negative effect. Phosphoric acid might react irreversibly with the zirconium dioxide surface, creating a layer of zirconium phosphate that might inhibit adhesion of the primer to the ceramic. Another explanation is the difficulties in eliminating acid residues from the cementation surface (127).

Air borne abrasion

To achieve a durable bond between a ceramic restoration and cement, it is necessary to enlarge the cementation surface for micromechanical retention (24, 55). With unmodified oxide ceramics the etching technique is insufficient to accomplish this. Techniques to increase the surface roughness, and consequently the bond strength, of oxide ceramics are often restricted to machining, grinding or airborne particle abrasion. Air borne particle abrasion with Al_2O_3 particles is the most widely used surface treatment method in dentistry and is considered an effective method of achieving micromechanical retention in oxide ceramics to the cement or to the veneering porcelain (5, 55, 56, 90).

The literature suggests two main retentive mechanisms for stronger bonding between ceramics and adhesive cement systems. First, if a pore is close to the surface, the material between the pore and the surface may break during sandblasting, creating a surface structure that enhances mechanical interlocking (128). Second, sandblasting removes contaminations from the surface, which also increases bond strength (94, 125). Sandblasting also has disadvantages, as it may tear the surface and cause surface damages, grain pullout and material loss (57, 76, 83-85). If such flaws induce surface cracks, they could act as fractural impressions and cause an insidious weakening of the ceramic (129). In the case of Y-TZP, a local phase transformation may occur which initially increases fracture strength but lacks transformation toughening over time, reducing the strength (84, 85, 123, 130, 131).

In Study I the blocks were blasted with $110\mu\text{m}$ Al_2O_3 particles for 10 seconds with an air pressure of 5 bars and a distance of 100 mm, with gentle movements of the blasting nozzle perpendicular

to the surface, in accordance with the manufacturer's instructions. Some studies have suggested that blasting ceramic surfaces with 50 μm particles would allow sufficient micromechanical retention (20, 63, 87, 94, 132). Results from other studies, however, indicate that a smaller particle size, such as 50 μm , polishes rather than enlarges the surface (133, 134). Several investigations have tested air pressure between 2.5 and 2.8 bars at a distance of 10 mm between the blasting tip and the object (20, 63, 87, 94, 132). Having an air pressure of 5 bars may have influenced the results by creating a rougher surface and a larger area that might improve the bond strength. On the other hand using ≥ 4 bars air pressure when sandblasting may tear the surface and induce flaws and cracks (131, 135, 136). By having a distance of 100 mm however, the force of sandblasting may be similar to the force applied when using lower air pressure closer to the surface. In study IV the specimens were sandblasted with 110 μm Al_2O_3 particles for 10 seconds with an air pressure of 2 bars at a distance of 10 mm, with gentle movements of the blasting nozzle perpendicular to the surface, in accordance with the manufacturer's instructions. The findings of the systematic review also supported this methodology.

Surface modification with additive impaction technique

Both the surface modification mediums tested in Study II, glass granules and polymer granules, were embedded in the ceramic surface during green-stage pressing, thus aiming to create a basis for a surface structure suitable for bonding (98). When using glass granules embedded prior to sintering, the glass will melt and partly remain on the surface after sintering. This glassy surface must be removed prior to cementation, which is done by etching the surface with HF. The possible residues of glass left on the cementation surface may enhance the chemical retention between the glass-containing zirconium dioxide surface and the adhesive cement system, thus increasing bond strength in addition to the micromechanical retention created by etching. Surface modification may also affect the physical properties of the Y-TZP material, especially if phase transformation of the crystals occurs in the material. (84, 85, 123, 130, 131, 137, 138). Study IV further investigated these issues of surface modification.

When polymer granules are used, the granules burn out during sintering, leaving a surface structure with ready-made micromechanical retention. The burn-out process is not clean, however, since contaminants could be seen on the treated surface after sintering. Those contaminants may have remained after etching with HF, which could affect subsequent bonding. The results in this study however, did not verify this assumption. The choice to investigate only the glass medium in Study IV was because it likely enables chemical retention, while the polymer medium only provides micromechanical retention.

Cementation process

All *in vitro* studies used the same cementation procedures, in accordance with the suggested methods of the manufacturer. Using an alignment apparatus with an applied seating load of 15 N during polymerization ensured that the axes of the specimens were perpendicular and that the load for all specimens was standardized during polymerization (139). The cement was light-cured with different curing lamps in each study. Each study used the curing lamp available at the facility at the time of the study. The polymerization light intensity in Study I was 300 mW/cm² (mean light intensity) and the curing time was 40 seconds in each of four directions, 90° apart, and a final 60 seconds in one direction with the seating load removed. In Study II, the polymerization light intensity was 1100 mW/cm² and in Study IV, 1000 mW/cm². In both these studies, the curing time was set to 20 seconds in each of four directions, 90° apart, and a final 60 seconds with the seating load removed. An overly short curing time may give the impression that the cement has completely set when only the outermost has initially hardened, and thus fail to fully utilize the properties of the cement system (121). The sufficient light intensity and curing time for each study was determined according to the manufacturer's recommendations

The maximum accepted clinical value for cement thickness of adhesive cement systems has been debated for many years. In theory, the thickness should be from 30 to 50 µm, but clinical studies of accuracy, combined with longevity data, indicate that the cement may seal wider internal gaps between the tooth and restoration and function for many years. Improper cement thickness, may trigger

technical or biological complications that limit the durability of a restoration (6, 25). No measurements of cement thickness were made which means that this is an unknown variable. However, because all specimens underwent the same cementation procedure, there was likely little variation in cement thickness between specimens treated with the same cement system.

Artificial aging process (Water storage, TC, mechanical pre-load)

It is generally acknowledged that environmental influences in the oral cavity can affect the durability of the bond strength. Artificial aging processes attempt to mimic the degradation that orally placed restorations undergo by decreasing the strength of the materials and their bond (56, 57, 64, 93, 140, 141). There is, however, no consensus regarding an appropriate procedure for aging (93, 140). Studies I and II conducted artificial aging using TC. Between tests, Study I specimens were stored in water at 37°C while Study II specimens were stored in water at RT (approx. 20°C). This water storage was primarily to prevent desiccation of the cement and was not aimed to be an artificial aging process. TC is preferred when the interfaces can be influenced by thermal stress and reduction in bond strength can be observed (24, 58, 64, 142). However, the extent to which TC mimics clinical situations has been questioned. Temperature (5°-55°C), dwell time, and the size, number and materials composition of specimens in the TC are also some topics that have been discussed in the literature (57, 140). The difference of approximately 50°C between the two baths may not be great enough to affect the ceramics, but affect the interfaces of the included materials (141). Nevertheless, a wet environment may affect the ceramics, which are susceptible to mechanical degradation in the presence of water when combined with stress corrosion at a crack tip. Fatigue and subcritical crack growth reduces the strength of the material and can result in fracture loads that are lower than the original strength of the material (55, 143). Other artificial aging methods, such as fatigue with dynamic cyclic loading, have been suggested and tested when evaluating bond strength (57, 114, 144).

The dynamic cycling fatigue method, or mechanical preload, is preferred when evaluating the flexural and fracture strength of a material (143, 145). In fatigue testing, it is important to describe the

applied load, the number of cycles, and the rate of application of the load. Study IV achieved its fatigue process by cyclic preloading in a wet environment, followed by TC, which are accepted and commonly used methods in *in vitro* studies (93, 131, 143, 145). Dental ceramics are susceptible to slow crack growth, and cements, to degradation. During cyclic preloading, cracks tend to propagate even under a small load due to stress corrosion at the crack tip (143, 145). The applied load and the time of the test cycle can vary (131). The load interval used in Study IV was 10 to 100 N. This interval was chosen to avoid the risk of fracture during preload.

The choice of tests

Bond strength definition and tests

Bond strength can be tested using various test methods, each with advantages and disadvantages. Recommendations for choice of adhesive cementation of oxide ceramics and adhesive bonding systems are mainly based on *in vitro* tests (117, 118, 146). These test methods vary and include tensile/micro-tensile (TBS/ μ TBS) bond strength test, shear/micro-shear (SBS/ μ SBS) bond strength test, and pullout tests (61). The most commonly used methods for testing bond strength, including micro-tensile and micro-shear tests (120, 146). Variations in bond strengths are large, with mean values ranging from debonding to above 50 MPa, and depend on the chosen test method. Results are not directly comparable, but they give an indication on the obtained bond strength (117, 120, 146). Studies I, II and III defined bond strength as clinically sufficient, regardless of the test method used, when the values were ≥ 20 MPa. From a clinical perspective, it is difficult to set a standard and define what constitutes sufficient, adequate, and durable bond strength. The value of 20 MPa has proved clinically sufficient, compared to the lower limit of the bond strengths between resin and enamel (100).

The advantage of the SBS test are that it is suitable for evaluating adhesive and restorative materials and is considered to be a user friendly test method (120). The test's reliability, however, is questionable (117, 119, 120). Stresses developing at the bonded interface have been shown to be more complex than the calculated load at failure. In the shear test, stresses close to the loading area are much higher and more complex than pure shear load. The test load

involves compressive as well as tensile stresses (117, 119, 120). The SBS test generally shows lower bond strength values than the TBS tests (118).

The advantage of the TBS test include that little material is consumed when the test is performed and that the stresses may be less complex, that is, allowing for a more even distribution of stress. One of the drawbacks is the production of the specimens and attachment during testing, since it may produce uneven stress distribution and affect the results (120). The use of microspecimens and the μ SBS or μ TBS tests have been developed to overcome these drawbacks (147). Results from previous studies indicate that a smaller bonding area provides higher bond strength values (117, 118, 120). This can be explained by the Weibull distribution, which shows that an increase in bond area increases the probability of encountering strength-limiting flaws and that a specimen's flaws are size dependent (116).

There are many different testing parameters, with different designs and materials of crosshead and loading conditions (117, 118). Studies I and II used a crosshead shaped like a knife-edge chisel for their SBS test to evaluate bond strength. The knife-edge introduces a bending moment and, in cylinders made of low modulus material, failure typically starts at the load site (148, 149). The interface between the ceramic, the adhesive cement system and the composite does not deform plastically to any great extent during such tests. In Studies I and II, the cylinders were made of a high modulus ceramics, where a bending moment also was present, but deformation during loading will occur in the cementation interfaces (149). The use of a knife-edge chisel crosshead concentrates stress at the load application area. Crossheads with a wire loop design have shown better stress distribution at the edge of the bonding area. There is other crosshead design as well, such as a notched-edge shear bond strength test that may produce more even stress distribution at the interfaces. Stress concentration with the use of the chisel may explain the small areas of cohesive failure that was found close to the loading point. It may also explain the lower bond strengths when the chisel crosshead is compared to crossheads with larger contact areas. Crosshead speed does not seem to influence bond strength values (120). The crosshead speed used, was within the limits suggested by the ISO/TS 11405:2003 (96).

Types of Failure

Study I provided a workable definition of the different types of failure. Adhesive failures occur between the different interfaces e.g. ceramic-cement-ceramic. Cohesive failures occur within either the ceramic materials or cement systems. In mixed failures, both adhesive and cohesive failures are found on the bonded area (117). In Studies I and II, the type of failure in debonded areas was determined by whether or not the ceramic surface was free of cement. However, classifying types of failure is difficult, especially for mixed failures. Classification of failure type remains an issue as there is no uniformly accepted classification system (118).

Surface analysis and characterization

Microscopy. In the *in vitro* studies (I, II and IV), the cementation surfaces of the specimens were examined under a light microscope during every step of the fabrication process. This examination checked if there were any remnants or other particles, such as dust, and whether the surface looked homogenous. Studies I and II also used light microscopy to perform fracture surface analyses of the bonded areas.

Interferometry (IFM) and Atomic force microscopy (AFM).

Topographical analysis using IFM and AFM characterized the surface roughness at the micrometer level (Studies II and IV) and nanometer level (Study IV) (42, 48, 62, 99, 118). Neither IFM nor AFM require specific sample preparation and neither are destructive in comparison to SEM (42). There are several parameters available, and three were selected according to the proposed guidelines for biomaterial surface characterization (99). The surface modification technique aiming to modify the surface at micrometer level would likely affect topography at the nanometer level as well. Therefore the surfaces were examined using both IFM and AFM.

Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). In addition to characterizing the surface topography at the micrometer and nanometer levels, it was also performed a morphological examination in Study IV that used SEM in combination with EDS to describe the atomic composition.

X-ray diffraction (XRD). In Study IV, XRD was performed to analyze and identify crystalline structure. Using EDS and XRD technologies made it possible to analyze the chemical composition of the ceramic surface and detect any remnants of the impaction medium or possible phase transformations (tetragonal → monoclinic) in the Y-TZP that could weaken the material (123, 131, 137, 138).

Biaxial flexural strength test- load until fracture

One possible drawback of the surface treatments is that mechanical properties, such as strength, of the oxide ceramics may be affected. The impaction-modified technique may leave remnants of the impaction medium and induce production-related flaws that may act as fractural impressions (82-85). This was one of the issues investigated and evaluated in Study IV using a biaxial flexural strength test, conducted in accordance with ISO 6872:2008 (18). This type of test can, in a controlled environment, provide information on basic mechanical properties, such as fracture strength, and compare different materials. Flexural strength can be measured in a three-point flexure test, a four-point flexure test, or a biaxial flexure test. In all cases, the load applied increases until fracture. The values of flexural strength obtained with the four-point flexure test are generally lower because surface cracks or critical flaws are more likely to develop between the two loading pistons than in the more limited area of a three-point flexure test. In the biaxial flexure test, in which a disc is loaded in the center, the probability of edge failure, which also depends on surface finish of the specimen, is reduced compared to three- or four-point flexure tests (41, 145).

Methods: Systematic review

Systematic literature reviews should systematically collect and summarize data, and provide results and conclusions in a transparent way based on well-defined questions, and set criteria, protocols, and evaluation processes (116, 150). Lack of consensus was found in *in vitro* studies evaluating bond strength regarding the appropriate testing methodology, interpretation of result, and application of clinical outcomes. Shared protocols for *in vitro* studies could ease comparison between study results (116, 118, 120).

Study design

In a literature review, it is important that the aims and questions are well defined. This makes inclusion and exclusion criteria easier to set, streamlines summary of results, and allows more meaningful conclusions. To address the aims and questions of the literature search, Study III sought literature on all possible surface treatments of oxide ceramics to promote bonding and the bond strength results from different test set-ups. The inclusion criteria in Study III were set to include oxide ceramics, with no consideration given to the adhesive cement system used or whether the materials and surface treatments were commercially available or purely experimental. These criteria made a general inventory possible.

To address Question 2 (Do any of the methods provide sufficient bond strength for the retention of dental restorations without the need for macromechanical retention?), it included studies on artificial aging and evaluating the ceramic surface before and after bond strength testing. Initial bond strength tests often show higher strength values than after artificial aging (56, 64). If no artificial aging has been conducted, then it is difficult to evaluate and apply the bond strength values to bond strength *in vivo*. The surface analysis and the failure types are also an evaluation of the bond strength test method, and light microscopy analysis only was not considered sufficient (117, 118).

The literature search was limited to only one database, PubMed. It may have been advantageous to perform the literature search in other databases, such as ScienceDirect (Elsevier), as well. Furthermore, each of the two aims and questions should preferably have been answered by two separately literature searches. Clinical relevance is lacking because the included studies are all *in vitro* studies. Advantages of Study III include its design: two investigators, calibrated in accordance with set criteria, conducting the literature search, reviewing the literature, and choosing the studies to include.

Results

The results of the systematic review showed that there is a wide variety both of surface treatments for oxide ceramics and of test methods that evaluate the bond strength between oxide ceramics and adhesive cement systems. This makes it difficult to compare studies and make recommendations of optimal surface treatment of oxide ceramics.

Surface treatments

Seven categories of surface treatments were defined from the literature: as-produced, grinding/polishing, airborne particle abrasion, surface coating, laser treatment, acid treatment, and primer treatment. Each surface treatment category included several specific surface treatments, some of which are commercially available and others experimental. In the included studies, three surface treatments showed bond strength values exceeding 20 MPa: airborne particle abrasion with aluminum oxide particles; (28, 72, 94, 104, 108, 110-113) airborne particle abrasion in combination with silica-coating, such as tribochemical silica-coating; (72, 89, 94, 101, 106, 112, 114) and a primer in combination with an abrasive or silica-coating surface treatment (28, 94, 106, 108, 115) (Table 12). The results from Study III indicate that micromechanical retention is necessary to achieve a durable bond between oxide ceramics and an adhesive cement system.

In almost all the studies included in Study III, airborne particle abrasion used aluminum oxide particles with a particle size ranging from 50 to 125 μm . This may be because it is an accessible, time-efficient, and economical method to use (55). Several studies show that it is an effective cleaning method and influences higher bond strength (125-127, 151, 152). Alternative airborne abrasion particles, such as synthetic diamond particles, have also been evaluated and show high bond strength values (112).

Silica-coating, more specifically tribochemical silica-coating, suitable for glass-infiltrated oxide ceramics, but it appears to be less suitable for oxide ceramics such as aluminum oxide and stabilized zirconium dioxide, resulting in reduced bond strength after artificial aging (33, 55). However, in Study III, surface treatment with tribochemical silica-coating showed sufficient bond strength values, regardless of test method (72, 89, 94, 101, 106, 112, 114). This indicates that tribochemical silica-coating also was a surface treatment that could be used to achieve a durable bond between oxide ceramics and adhesive cement systems.

The majority of the included studies used an adhesive cementation system that included a primer. The literature indicated that there is interest in finding a mechanical or chemical surface treatment that combines with a primer (55). The rapid development of new adhesive materials means that there are a variety of primers and

various ways of applying them to the cementation surfaces (91). The components and compositions of the primers found in the included studies were not record.

The production process of the glass-modified surface in Studies II and IV did not seem to induce any of the surface damage that has been identified after machining or after airborne particle abrasion. The modified surfaces showed irregularities, probably as a result of uneven distribution of the medium prior to pressing. This implies that the coating procedure could be refined, though the bond strength achieved already appears to be sufficient. When using the impaction-modified technique, the cementation surface becomes more comparable to glass ceramic. Even if microcracks and flaws are present after sintering, they are likely reduced by the etching procedure. HF seems to dissolve the glass components and produce a more smoothed, but still irregular surface, with both micromechanical and chemical retention that facilitates adhesion of the adhesive cement (24, 93).

In Study IV the cement remained intact on the glass-modified surface after the artificial aging procedure, indicating a stable bond between Y-TZP and adhesive cement. Furthermore, the results show that the cement had an enhancing effect, probably due to the sealing of the surface, reinforcing the material (11). Also, after the artificial aging procedures, the unmodified Y-TZP and the sandblasted Y-TZP showed areas with loose cement and air pockets throughout the cementation surface. This was not found in the glass-modified Y-TZP groups. These findings suggest that the cementation surface of the restorations needs a surface treatment that enhances more than just micromechanical retentions (138). The benefit of the modified-additive production technique is that it seems to achieve both micromechanical and chemical retention, producing durable bond strength between the surface-modified Y-TZP and adhesive cement system.

Shear bond strength: How does surface treatment affect bond strength?

The mean values for the strength test from the studies (n=23) included in Study III, range from debonding to 45 MPa for the SBS test, from 8 to 26 MPa for the μ SBS test, from debonding to 38 MPa for the TBS test, and from debonding to 53 MPa for the μ TBS test (Table 12). It is difficult to make any statements based on the results

of the studies because different materials were tested with differing methods and scattered results.

Even though the results from reviewed studies with the same design and materials as Studies I and II were widely spread, the achieved results fall within a same range (76, 78, 89, 101, 102, 106, 107, 112). The values achieved for Variolink® II and Panavia™ F in Study I agree with other studies (94, 113) and indicate that these might be sufficient for adhesive cementation. Some clinical studies have used these cement systems for adhesive cementation of densely sintered aluminum oxide or Y-TZP (153-156). In Study II the bond strength in the unmodified groups was reasonably high (18-20 MPa), considering that no surface modification was done. But bond strength in the surface-modified groups was higher, regardless of which cement that was used. The cementation surface that was modified with glass granules and cemented with Variolink® II showed the highest mean bond strength (35 MPa). This suggests that it may be a glassy phase left on the glass-modified Y-TZP surface, as for feldspathic porcelain and glass ceramics, that enhances chemical bonding between the cement and the ceramic surface (104, 110).

The results from Study II are sufficient for clinical use if compared to conclusions reached in previous studies, where the values for SBS range between 15 and 30 MPa (76, 78) . Even though it is not possible to compare numerical values from different studies, the results, if restricted to shear bond strength only, support the use of the investigated method for restorations such as veneers, onlays and RBCBs. Clinical studies, however, are needed to confirm the findings of *in vitro* studies. There are limited data on the clinical outcome of adhesive bonding to oxide ceramics (32) and only a few studies on oxide-based RBCB (54). Independent of the adhesive cement system, Sasse et al. showed promising results, during the 3 first year with a small number (n=25) of single-retained stabilized zirconium dioxide- based RBCBs. (53).

Types of Failure

When there are cohesive failures in the substrate materials, the validity of the reported bond strength is questionable. Only adhesive failures or mixed failures with small areas (less than 10%) should be considered (117, 118). Cohesive failure is explained by the

mechanics of the test and the brittleness of the materials used and is more frequent for macro- than micro-bond strength tests (118, 120).

In Study I, the predominant failure type of some cements (Variloink® II, Bisco Illusion™, RelyX™ Veneer) was adhesive at the untreated surface. It is possible that higher bond strength could have been achieved if the untreated surfaces of the cylinders had been treated as the blocks. For the remaining groups (Bisco Choice™, Nexus™ 2 and Panavia™ F) the failure type was adhesive at the treated surface or at both surfaces. It may be that surface treatment is irrelevant for some adhesive cement systems and important for other systems. The cohesive failures in Study II can be explained by tensile stress in the feldspathic porcelain that caused a propagation of the crack into the substrate, leading failure in the feldspathic porcelain (120). The surface-modified groups showed less debonding between the cement and the Y-TZP surface than the two control groups, where all the failures were adhesive. It is noteworthy that the groups with adhesive failures had lower bond strength and surface roughness.

The classification of the various failure types, in particular complex failures, might have been more accurate if the examination had been done under greater magnification, such as SEM-analysis, revealing remnants that are undetectable with microscopy (117). It may be that the fracture types in Studies I and II were sometimes more complex than classified. If that is the case, the measured values are dependent on the mechanical strength of the cement rather than the bond strength between oxide ceramic/cement or cement/oxide ceramic or feldspathic porcelain.

Shear bond strength: How do adhesive cement systems affect bond strength?

The use of adhesive cement systems including a primer appears to have improved bond strength in Study I. Coupling agents such as adhesive cement consist of bifunctional molecules that function between the inorganic ceramic surface and the organic polymer matrix of the resin. The lack of glass or silica-containing matrix on the material unable the chemical bond created by silanization (55, 57). The exact bonding mechanisms of silane to oxide ceramics are still unclear. A durable bond between densely sintered aluminum oxide or stabilized zirconium dioxide and adhesive cement systems

(combined with their corresponding primers) can be achieved, especially with MDP-containing adhesive cement systems (20, 58, 64, 94, 157). Some authors recommend that adhesive cements containing MDP can be used without primer (87), whilst others suggest that the use of a primer enhances the wetting of the surface and increases the cooperative interaction between the interfaces (20, 58, 93-95). All *in vitro* studies (I, II and IV) used the corresponding primers for each cement system. The use of a primer and its action as wetting agent, may explain some of the results in Study I, where the MDP-containing cement Panavia™ F, and Variolink® II, a Bis-GMA-based cement, had the highest bond strength values, irrespective of pretreatment (20, 63, 94, 158). Of the six different bonding systems, only two Nexus™ 2 and RelyX™ Veneer, decreased significantly ($p < 0.05$) in bond strength after TC. Variolink® II showed higher bond strength after TC, where TC may have acted as a catalyst and promoted continued curing of the adhesive cement. A possible explanation of the low bond strength values for the adhesive cement system RelyX™ Veneer may be that it is a one-paste system that is light cured only. If the aluminum oxide cylinders partly hindered light from reaching the cement, there might have been poorly cured areas. The quality of the transmitted light could have influenced the conversion and, consequently, bond strength (121). Differences in compositions between the various cements, such as filler particles and other chemical additives, could also have influenced the level of conversion (121).

Biaxial flexural strength: How does surface treatment affect flexural strength?

The results for flexural strength of unmodified and sandblasted Y-TZP in Study IV were in accordance with ISO values for the substructure of non-adhesively cemented restorations (18), but lower than in other studies (41, 51, 131, 137, 159). The specimens were fabricated with uniaxially applied force and the results are not directly comparable with other studies due to variations in study design, such as the material tested, fabrication process, use of artificial aging, and loading conditions (41, 51, 131, 159). Glass-modified Y-TZP showed a decrease in flexural strength compared to the unmodified groups. The presence of a low-modulus glass phase on the surface

of Y-TZP could explain the results in Study IV. Glass-infiltrated aluminum oxide and zirconium dioxide have shown comparable results, where the glass matrix will give lower mechanical properties (41, 47). The groups submitted to etching before sintering showed higher values than the groups submitted to etching after sintering. This may be because the glass medium that was etched away before sintering enabled the grains to sinter together. In the group with etching after sintering, the glass medium seemed to prevent sintering, leaving voids between the grain boundaries, which weakens the material. The results from the surface analysis in Study IV amplify this assumption, with voids/porosities apparent in the SEM images. Comparing the unmodified and modified Y-TZP, the group that was sintered and sandblasted showed the highest values, with an increase in flexural strength when cement was included. Sandblasting may have induced small amount of stress concentration at the surface, causing phase transformation (83, 130, 131, 136, 160, 161) and resulting in compressive stress that increased flexural strength (93, 130, 159-162). Sandblasting of the Y-TZP surface seems to provide high initial value, (130, 131), but further investigations of how the material will behave over time are needed.

Surface roughness and chemical surface composition:

How does the surface treatment affect material composition and properties?

The combined use of IFM, AFM, SEM and XRD analyses is a procedure employed to characterize a surface and the effect of surface treatments (42, 48, 62, 131, 136, 137, 163). In Study II, the analysis of the cementation surfaces made by microscopy and IFM showed that each of the surface-modified groups had a surface with increased roughness after sintering followed by etching in comparison to unmodified Y-TZP. Study IV found similar results when AFM analysis was also included. Unmodified Y-TZP showed no change in microstructure, either before or after the sintering and etching procedure. Airborne particle abrasion increased surface roughness, creating microretentive grooves, but the surface also showed a damaged and deformed surface as found in other studies (84, 85, 123, 131, 136, 160). The SEM analysis exposed defects (i.e., voids/porosities) likely caused by the fabrication technique.

Most of the fabrication techniques and every type of ceramic have varying degrees of defects. The material should be handled carefully to minimize these defects (48, 85). In the groups with glass modification, the degree of sintering of the ceramic powder and the formation of grain boundaries were different than for unmodified Y-TZP. The glass between the grain boundaries prevented the grains from sintering together, resulting in a porous material. This may have happened because the glass melted during sintering and settled in the intergranular areas. After the sintering process is complete, it forms a void/porosity (50, 123). The presence of voids on the surface increases surface roughness, which enhances micromechanical retention. On the other hand, voids might also weaken the material, depending on the location, size, shape and distribution of such defects (47, 85, 123, 138).

Based on the XRD analysis results, two crystalline structures, monoclinic and tetragonal, were predominant. Monoclinic phase was identified after sandblasting the unmodified Y-TZP and even more monoclinic phase in the glass-modified Y-TZP. This indicates that, when glass is present, the Y-TZP becomes unstable and phase transformation occurs (47). The same applies for the sandblasted Y-TZP surface, although not in the same degree. These findings agree with other studies (84, 131, 135, 159).

Clinical significance

There are few clinical trials available on adhesively cemented oxide-based ceramic restorations and their conclusions differ as to whether they are a valid alternative to metal frameworks (32, 164, 165). According to a systematic review, the survival rate of tooth-supported and implant-supported stabilized zirconium dioxide-based crowns is comparable to PFM crowns. One of the most common reasons for technical failures or complications of tooth-supported crowns was loss of retention (165). However, there is often no information available on the possible causes of loss of retention, and several different cement systems have been used in the trials, which makes it difficult to draw general conclusions.

The development and use of oxide ceramics and adhesive cement systems may provide an extended treatment option with tooth-supported crowns and FDPs. Possible indications for use of adhesive cementation techniques with oxide ceramics are for tooth preserving

treatment options, such as RBCBs, or minimally invasive treatments in clinical situations with limited retention, such as moderate tooth wear. (53, 54). In those cases the risk for unnecessary preparation of the tooth and surrounding tissues is less. It may also benefit young patients who are not yet eligible for implant treatment (53).

When choosing cement system, a dual or chemically curable adhesive cement system may be the best choice. Several studies suggest that MDP-containing adhesive cement systems enhance the bond strength between oxide ceramics and cement, with or without any surface treatment (55, 58, 59, 95, 163). Well-design longitudinal studies with large patient groups are required before extending the indication of treatment options.

Future investigations

Commercially available materials are usually tested according to a set of standard tests to evaluate their suitability and performance as dental materials. These standards are developed and published by the International Organization for Standardization (ISO). The most common test methods are *in vitro*, but *in vivo* studies are needed to make clinical recommendations and statements. Test set-ups differ currently, so the development and standardization of test methods is an important component of future research. Studies that investigate the effect of different artificial aging process on the various materials and interfaces, that improve categorization of failure types, and that validate findings for a reliable clinical application will all be necessary in the future.

All-ceramic dental restorations have improved considerably in their performance, and Y-TZP is the one with most preferable material properties (5). Still, there is much to learn about oxide ceramics and production of restorations. The main focus of development of surface treatment for oxide ceramics seems to be for stabilized zirconium dioxide. At present, the development of translucent Y-TZP and its application could be of great interest (35). The basic composition between traditional oxide ceramics and translucent Y-TZP is similar (45), which means that the surface treatments that enhance the adhesive bond between oxide ceramic and adhesive cement system work for booth materials. Before making clinical recommendations regarding glass-modified Y-TZP, it is necessary to conduct further clinical studies to investigate and evaluate both the influence of the

medium on the cementation surface and its mechanical properties throughout the entire fabrication process of the restoration. Any surface modification or pretreatment can affect the properties of the material, and those effects should be assessed.

CONCLUSIONS

Sufficient bond strength can be achieved to densely sintered aluminum oxide by using a Bis-GMA based dual-cured resin cement system or a phosphate-monomer containing resin cement system. (Study I)

Recommendations on how to achieve a sufficient bond between densely sintered aluminum oxide and adhesive cement system should be based on the adhesive cement system used since bond strength will depend on the specific system. (Study I)

The surface structure and the chemical composition of Y-TZP ceramics produced using a glass-modification technique differ from that of unmodified Y-TZP. The differences include a rougher surface structure, superficial glass remnants and the presence of monoclinic phase. (Study II and IV)

Etching the glass-modified Y-TZP before sintering creates a more homogenous surface than etching after sintering. (Study IV)

Impaction modification with either glass granules or polymer granules in an additive production technique can create a bondable cementation surface suitable for Y-TZP-based restorations with a shear bond strength exceeding 20 MPa. (Study II, IV)

As-produced oxide ceramics needs to be surface treated to create durable bond strength. (Study III and IV)

Airborne particle abrasive surface treatment, with or without the use of primer treatment, can create sufficient bond strength for the bonding of oxide ceramics. The same applies for treatment with silica-coating, but only with the use of a primer treatment. This conclusion, however, is not yet confirmed by clinical studies. (Study III)

For each specific ceramic material, consideration should be given to surface treatment and choice of adhesive cement system in order to achieve sufficient bond strength. No general recommendations can be given. (Study III)

The flexural strength of Y-TZP decreased after glass modification, but increased after cementation. Further studies are needed before recommending the technology for clinical applications. (Study IV)

ACKNOWLEDGEMENTS

This thesis would not have been possible without all the inspiring and generously supporting people around me. I wish to express my sincere gratitude to **everyone**, and in particular I wish to thank:

Associate professor Per Vult von Steyern, my supervisor; for your expert guidance in academic thinking, writing and performing. With your never-ending enthusiasm and patience, you have motivated me during all my years on the faculty to start and complete this work and to continue the journey.

Professor Ann Wennerberg, my co-supervisor; for sharing your knowledge with me and for encouraging and guiding me. Thank you also for always making me feel welcome.

Associate professor Erik Strandman; who encouraging me to start the journey.

Professor emeritus Krister Nilner; for always making time for me and my questions, and giving me wise advice.

My co-authors:

Johan Zethreus and Per-Åke Ransbäck; for your catching curiosity and contribution.

Christel Larsson; for sharing your knowledge and for your engagement. I look forward to future collaborations and challenges.
Madeleine du Toit; for sharing your knowledge, for your curiosity

on life, and for your engagement, from small issues to large, but above all for being a real friend.

Associate professor Ryo Jimbo; for sharing your knowledge, for all your support, and making me feel welcome and that I was one of you, at work and in our spare time. I look forward to future collaborations.

Bruno Chrcanovic and Associate professor Martin Andersson; for sharing your knowledge, for your hard work and contribution.

Professor Ulf Örtengren; for valuable comments on the research program during my half-time seminar.

All my dear colleagues, past and presents, at the Faculty of Odontology, Malmö University, and especially the entire staff of the Department of Materials Science and Technology and the Department of Prosthodontics; thank you for all your help and care.

Among dear colleagues I would like to send a special thank you to: Lars Olsson, Zdravko Bahat, Camilla Johansson and Ewa Linderoth; for taking the time to support me and follow along on my journey, and also for sharing your knowledge, work-related or not. Especially Camilla and Lars for cheering me on during the hard times with your notes, and for watching the TC. Håkan Fransson; for helping me to check, re-check and double check as someone wise taught me.

All great women who have inspired me, especially Camilla Ahlgren, Christina Diego-Löfgren; thank you for sharing your experience and knowledge, for all your support, for sharing ups and downs and for being my friends. Kerstin von Bahr and Eva-Lotta Rymark; for your thoughtful and precious comments on all subjects that matters in life, and, of course all nice dinners!

Per Egevad; for valuable assistance with computer management of the references and making the systematic review figures understandable.

Jenny Widmark; for valuable help in the field of publication rights.

Ulf Persson, Daniel Bengmark and Jonas Finnhult, starting up as the group of “After Drill” which developed into great friendship; each of you, thank you for all your support, cheering me on and saving me in my panic - we will always have the Christmas baking spirit!

The staff at Serviceenheten: Peter, Roger, Ingvar, Rickard, Richard, Steve and Ingemar; for always making things work in time!

Thanks to all present and past students at the undergraduate education program in Dental technology; for your catching curiosity and questions that encourage one to seek greater knowledge.

My fantastic friends; for all your love and support, especially to my dear friend Caroline Linné Erixon; for our endless phone calls where we discuss issues of all kinds and your encouraging comments and advice.

My family, my brother Apostolos and his Kristina; for always being there for me and being real friends, giving me distance from my thoughts with insightful and encouraging advice. My parents; for their endless love and making me believe in myself;: Μαμμα, μπαμπα, σας ευχαριστω για την αγαπη και την υποστηριξη σας και ο,τι προσφερατε για να φτασω μεχρι εδω.

Research grants were received from:
The Faculty of Odontology, Malmö University

REFERENCES

- (1) Trulsson U, Engstrand P, Berggren U, Nannmark U, Branemark PI. Edentulousness and oral rehabilitation: experiences from the patients' perspective. *Eur J Oral Sci* 2002; 110: 417-424.
- (2) Ozhayat EB, Gotfredsen K. Effect of treatment with fixed and removable dental prostheses. An oral health-related quality of life study. *J Oral Rehabil* 2012; 39: 28-36.
- (3) Bagewitz IC. Prosthodontics, care utilization and oral health-related quality of life. *Swed Dent J Suppl* 2007; 185: 7-81.
- (4) Zarone F, Russo S, Sorrentino R. From porcelain-fused-to-metal to zirconia: clinical and experimental considerations. *Dent Mater* 2011; 27: 83-96.
- (5) Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. *J Prosthodont Res* 2013; 57: 236-261.
- (6) Milleding P. Traditional prosthodontic preparations. In: Nilner K, Karlsson S, Dahl B L (editors). *A textbook of fixed prosthodontics: the Scandinavian approach*. 2 ed. Stockholm: Gothia fortbildning; 2013: 186-204.
- (7) ShahrbaF S, van Noort R, Mirzakouchaki B, Ghassemieh E, Martin N. Fracture strength of machined ceramic crowns as a function of tooth preparation design and the elastic modulus of the cement. *Dent Mater* 2014; 30: 234-241.
- (8) Vult von Steyern P, Jonsson O, Nilner K. Five-year evaluation of posterior all-ceramic three-unit (In-Ceram) FPDs. *Int J Prosthodont* 2001; 14: 379-384.
- (9) Edelhoff D, Ozcan M. To what extent does the longevity of fixed dental prostheses depend on the function of the cement? Working Group 4 materials: cementation. *Clin Oral Implants Res* 2007; 18 Suppl 3: 193-204.
- (10) Goodacre CJ, Campagni WV, Aquilino SA. Tooth preparations for complete crowns: an art form based on scientific principles. *J Prosthet Dent* 2001; 85: 363-376.

- (11) Koutayas SO, Vagkopoulou T, Pelekanos S, Koidis P, Strub JR. Zirconia in dentistry: part 2. Evidence-based clinical breakthrough. *Eur J Esthet Dent* 2009; 4: 348-380.
- (12) De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005; 84: 118-132.
- (13) Vult von Steyern P. Dental ceramics in clinical practice. In: Nilner K, Karlsson S, Dahl B L (editors). *A textbook of fixed prosthodontics: the Scandinavian approach*. 2, ed. Stockholm: Gothia fortbildning; 2013: 205-222.
- (14) Anusavice KJ. Dental ceramics. In: Anusavice KJ (editor). *Phillips' science of dental materials*. 11th ed. St. Louis: Saunders; 2003: 655-719.
- (15) Richerson D. Time, temperature and environmental effects on properties. In: Richerson D (editor). *Modern ceramic engineering: properties, processing, and use in design*. 3rd ed. Boca Raton, FL: CRC Taylor & Francis; 2006: 243-283.
- (16) Richerson D. Design approaches. In: Richerson D (editor). *Modern ceramic engineering: properties, processing, and use in design*. 3rd ed. Boca Raton, FL: CRC Taylor & Francis; 2006: 581-594.
- (17) Vult von Steyern P, Ebbesson S, Holmgren J, Haag P, Nilner K. Fracture strength of two oxide ceramic crown systems after cyclic pre-loading and thermocycling. *J Oral Rehabil* 2006; 33: 682-689.
- (18) International Standards Organization. *ISO 6872:2008 Dentistry - Ceramic materials* Geneva, Switzerland: European Committee for Standardization; 2008.
- (19) McLean JW. Evolution of dental ceramics in the twentieth century. *J Prosthet Dent* 2001; 85: 61-66.
- (20) Blatz MB, Sadan A, Arch GH, Jr, Lang BR. In vitro evaluation of long-term bonding of Procera AllCeram alumina restorations with a modified resin luting agent. *J Prosthet Dent* 2003; 89: 381-387.
- (21) Kern M, Sasse M, Wolfart S. Ten-year outcome of three-unit fixed dental prostheses made from monolithic lithium disilicate ceramic. *J Am Dent Assoc* 2012; 143: 234-240.
- (22) Fischer J, Stawarczyk B, Hammerle CH. Flexural strength of veneering ceramics for zirconia. *J Dent* 2008; 36: 316-321.
- (23) Holand W, Schweiger M, Watzke R, Peschke A, Kappert H. Ceramics as biomaterials for dental restoration. *Expert Rev Med Devices* 2008; 5: 729-745.
- (24) Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003; 89: 268-274.

- (25) Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systematic review. *J Prosthet Dent* 2007; 98: 389-404.
- (26) Rekow ED, Silva NR, Coelho PG, Zhang Y, Guess P, Thompson VP. Performance of dental ceramics: challenges for improvements. *J Dent Res* 2011; 90: 937-952.
- (27) Vult von Steyern P, al-Ansari A, White K, Nilner K, Derand T. Fracture strength of In-Ceram all-ceramic bridges in relation to cervical shape and try-in procedure. An in-vitro study. *Eur J Prosthodont Restor Dent* 2000; 8: 153-158.
- (28) Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. *J Dent Res* 2009; 88: 817-822.
- (29) Vagkopoulou T, Koutayas SO, Koidis P, Strub JR. Zirconia in dentistry: Part 1. Discovering the nature of an upcoming bioceramic. *Eur J Esthet Dent* 2009; 4: 130-151.
- (30) Manicone PF, Rossi Iommetti P, Raffaelli L. An overview of zirconia ceramics: basic properties and clinical applications. *J Dent* 2007; 35: 819-826.
- (31) Denry I, Kelly JR. State of the art of zirconia for dental applications. *Dent Mater* 2008; 24: 299-307.
- (32) Al-Amleh B, Lyons K, Swain M. Clinical trials in zirconia: a systematic review. *J Oral Rehabil* 2010; 37: 641-652.
- (33) Kern M. Resin bonding to oxide ceramics for dental restorations. *J Adhes Sci Technol* 2009; 23: 1097-1111.
- (34) Komine F, Blatz MB, Matsumura H. Current status of zirconia-based fixed restorations. *J Oral Sci* 2010; 52: 531-539.
- (35) Rinke S, Fischer C. Range of indications for translucent zirconia modifications: clinical and technical aspects. *Quintessence Int* 2013; 44: 557-566.
- (36) Andersson M, Oden A. A new all-ceramic crown. A dense-sintered, high-purity alumina coping with porcelain. *Acta Odontol Scand* 1993; 51: 59-64.
- (37) Andersson M, Razzoog ME, Oden A, Hegenbarth EA, Lang BR. Procera: a new way to achieve an all-ceramic crown. *Quintessence Int* 1998; 29: 285-296.
- (38) Oden A, Andersson M, Krystek-Ondracek I, Magnusson D. Five-year clinical evaluation of Procera AllCeram crowns. *J Prosthet Dent* 1998; 80: 450-456.
- (39) Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. *Biomaterials* 1999; 20: 1-25.
- (40) Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: an overview. *Dent Mater* 2008; 24: 289-298.

- (41) Yilmaz H, Aydin C, Gul BE. Flexural strength and fracture toughness of dental core ceramics. *J Prosthet Dent* 2007; 98: 120-128.
- (42) Deville S, Gremillard L, Chevalier J, Fantozzi G. A critical comparison of methods for the determination of the aging sensitivity in biomedical grade yttria-stabilized zirconia. *J Biomed Mater Res B Appl Biomater* 2005; 72: 239-245.
- (43) Chevalier J. What future for zirconia as a biomaterial? *Biomaterials* 2006; 27: 535-543.
- (44) Lughì V, Sergio V. Low temperature degradation -aging- of zirconia: A critical review of the relevant aspects in dentistry. *Dent Mater* 2010; 26: 807-820.
- (45) Jiang L, Liao Y, Wan Q, Li W. Effects of sintering temperature and particle size on the translucency of zirconium dioxide dental ceramic. *J Mater Sci Mater Med* 2011; 22: 2429-2435.
- (46) Kim MJ, Ahn JS, Kim JH, Kim HY, Kim WC. Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency. *J Adv Prosthodont* 2013; 5: 161-166.
- (47) Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II. Zirconia-based dental ceramics. *Dent Mater* 2004; 20: 449-456.
- (48) Casucci A, Mazzitelli C, Monticelli F, Toledano M, Osorio R, Osorio E, et al. Morphological analysis of three zirconium oxide ceramics: Effect of surface treatments. *Dent Mater* 2010; 26: 751-760.
- (49) van Noort R. The future of dental devices is digital. *Dent Mater* 2012; 28: 3-12.
- (50) Richerson D. Densification. In: Richerson D (editor). *Modern ceramic engineering: properties, processing, and use in design*. 3rd ed. Boca Raton, FL: CRC Taylor & Francis; 2006: 477-527.
- (51) Oh GJ, Yun KD, Lee KM, Lim HP, Park SW. Sintering behavior and mechanical properties of zirconia compacts fabricated by uniaxial press forming. *J Adv Prosthodont* 2010; 2: 81-87.
- (52) Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Y-TZP ceramics: key concepts for clinical application. *Oper Dent* 2009; 34: 344-351.
- (53) Sasse M, Eschbach S, Kern M. Randomized clinical trial on single retainer all-ceramic resin-bonded fixed partial dentures: Influence of the bonding system after up to 55 months. *J Dent* 2012; 40: 783-786.
- (54) Miettinen M, Millar BJ. A review of the success and failure characteristics of resin-bonded bridges. *Br Dent J* 2013; 215: E3.
- (55) Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? *Dent Mater* 2011; 27: 71-82.

- (56) Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater* 1998; 14: 64-71.
- (57) Mair L, Padipatvuthikul P. Variables related to materials and preparing for bond strength testing irrespective of the test protocol. *Dent Mater* 2010; 26: e17-23.
- (58) Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. *J Prosthet Dent* 2004; 91: 356-362.
- (59) Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. *Quintessence Int* 2007; 38: 745-753.
- (60) Derand T, Molin M, Kleven E, Haag P, Karlsson S. Bond strength of luting materials to ceramic crowns after different surface treatments. *Eur J Prosthodont Restor Dent* 2008; 16: 35-38.
- (61) Phark JH, Duarte S, Jr, Hernandez A, Blatz MB, Sadan A. In vitro shear bond strength of dual-curing resin cements to two different high-strength ceramic materials with different surface texture. *Acta Odontol Scand* 2009; 67: 346-354.
- (62) Casucci A, Osorio E, Osorio R, Monticelli F, Toledano M, Mazzitelli C, et al. Influence of different surface treatments on surface zirconia frameworks. *J Dent* 2009; 37: 891-897.
- (63) Awliya W, Oden A, Yaman P, Dennison JB, Razzoog ME. Shear bond strength of a resin cement to densely sintered high-purity alumina with various surface conditions. *Acta Odontol Scand* 1998; 56: 9-13.
- (64) Wegner SM, Kern M. Long-term resin bond strength to zirconia ceramic. *J Adhes Dent* 2000; 2: 139-147.
- (65) Gomes AL, Castillo-Oyague R, Lynch CD, Montero J, Albaladejo A. Influence of sandblasting granulometry and resin cement composition on microtensile bond strength to zirconia ceramic for dental prosthetic frameworks. *J Dent* 2013; 41: 31-41.
- (66) Inokoshi M, Kameyama A, De Munck J, Minakuchi S, Van Meerbeek B. Durable bonding to mechanically and/or chemically pre-treated dental zirconia. *J Dent* 2013; 41: 170-179.
- (67) Liu D, Pow EH, Tsoi JK, Matinlinna JP. Evaluation of four surface coating treatments for resin to zirconia bonding. *J Mech Behav Biomed Mater* 2014; 32: 300-309.
- (68) Baldissara P, Querze M, Monaco C, Scotti R, Fonseca RG. Efficacy of surface treatments on the bond strength of resin cements to two brands of zirconia ceramic. *J Adhes Dent* 2013; 15: 259-267.
- (69) Derand T, Molin M, Kvam K. Bond strength of composite luting cement to zirconia ceramic surfaces. *Dent Mater* 2005; 21: 1158-1162.

- (70) Aboushelib MN. Fusion sputtering for bonding to zirconia-based materials. *J Adhes Dent* 2012; 14: 323-328.
- (71) Usumez A, Hamdemirci N, Koroglu BY, Simsek I, Parlar O, Sari T. Bond strength of resin cement to zirconia ceramic with different surface treatments. *Lasers Med Sci* 2013; 28: 259-266.
- (72) Akyil MS, Uzun IH, Bayindir F. Bond strength of resin cement to yttrium-stabilized tetragonal zirconia ceramic treated with air abrasion, silica coating, and laser irradiation. *Photomed Laser Surg* 2010; 28: 801-808.
- (73) Akin H, Ozkurt Z, Kirmali O, Kazazoglu E, Ozdemir AK. Shear bond strength of resin cement to zirconia ceramic after aluminum oxide sandblasting and various laser treatments. *Photomed Laser Surg* 2011; 29: 797-802.
- (74) Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. *J Prosthet Dent* 2007; 98: 379-388.
- (75) Aboushelib MN, Matinlinna JP, Salameh Z, Ounsi H. Innovations in bonding to zirconia-based materials: Part I. *Dent Mater* 2008; 24: 1268-1272.
- (76) Phark JH, Duarte S, Jr, Blatz M, Sadan A. An in vitro evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface. *J Prosthet Dent* 2009; 101: 29-38.
- (77) Bottino M, Bergoli C, Lima E, Marocho S, Souza R, Valandro L. Bonding of Y-TZP to Dentin: Effects of Y-TZP Surface Conditioning, Resin Cement Type, and Aging. *Oper Dent* 2014; 39: e1-9.
- (78) Jevnikar P, Krnel K, Kocjan A, Funduk N, Kosmac T. The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics. *Dent Mater* 2010; 26: 688-696.
- (79) Lung CY, Botelho MG, Heinonen M, Matinlinna JP. Resin zirconia bonding promotion with some novel coupling agents. *Dent Mater* 2012; 28: 863-872.
- (80) Chen L, Suh BI, Brown D, Chen X. Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths. *Am J Dent* 2012; 25: 103-108.
- (81) Koizumi H, Nakayama D, Komine F, Blatz MB, Matsumura H. Bonding of resin-based luting cements to zirconia with and without the use of ceramic priming agents. *J Adhes Dent* 2012; 14: 385-392.
- (82) Luthardt RG, Holzhuter MS, Rudolph H, Herold V, Walter MH. CAD/CAM-machining effects on Y-TZP zirconia. *Dent Mater* 2004; 20: 655-662.
- (83) Zhang Y, Lawn BR, Malament KA, Van Thompson P, Rekow ED. Damage accumulation and fatigue life of particle-abraded ceramics. *Int J Prosthodont* 2006; 19: 442-448.

- (84) Guess PC, Zhang Y, Kim JW, Rekow ED, Thompson VP. Damage and reliability of Y-TZP after cementation surface treatment. *J Dent Res* 2010; 89: 592-596.
- (85) Denry I. How and when does fabrication damage adversely affect the clinical performance of ceramic restorations? *Dent Mater* 2013; 29: 85-96.
- (86) Kitayama S, Nikaido T, Ikeda M, Alireza S, Miura H, Tagami J. Internal coating of zirconia restoration with silica-based ceramic improves bonding of resin cement to dental zirconia ceramic. *Biomed Mater Eng* 2010; 20: 77-87.
- (87) Sadan A, Blatz MB, Soignet D. Influence of silanization on early bond strength to sandblasted densely sintered alumina. *Quintessence Int* 2003; 34: 172-176.
- (88) Matinlinna JP, Heikkinen T, Ozcan M, Lassila LV, Vallittu PK. Evaluation of resin adhesion to zirconia ceramic using some organosilanes. *Dent Mater* 2006; 22: 824-831.
- (89) Heikkinen TT, Lassila LV, Matinlinna JP, Vallittu PK. Effect of operating air pressure on tribochemical silica-coating. *Acta Odontol Scand* 2007; 65: 241-248.
- (90) Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dent Mater* 2007; 23: 45-50.
- (91) Amaral M, Belli R, Cesar PF, Valandro LF, Petschelt A, Lohbauer U. The potential of novel primers and universal adhesives to bond to zirconia. *J Dent* 2014; 42: 90-98.
- (92) Wada T. Development of a new adhesive material and its properties. *Proceedings of the international symposium on adhesive prosthodontics: Academy of Dental Materials, Amsterdam, The Netherlands; 1986: 9-18.*
- (93) Qeblawi DM, Munoz CA, Brewer JD, Monaco EA, Jr. The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. *J Prosthet Dent* 2010; 103: 210-220.
- (94) Hummel M, Kern M. Durability of the resin bond strength to the alumina ceramic Procera. *Dent Mater* 2004; 20: 498-508.
- (95) Tanaka R, Fujishima A, Shibata Y, Manabe A, Miyazaki T. Cooperation of phosphate monomer and silica modification on zirconia. *J Dent Res* 2008; 87: 666-670.
- (96) International Standards Organization. ISO/TS 11405:2003, Dental materials-Testing of adhesion to tooth structure. Geneva, Switzerland: European Committee for Standardization; 2003.
- (97) Derand P, Derand T. Bond strength of luting cements to zirconium oxide ceramics. *Int J Prosthodont* 2000; 13: 131-135.

- (98) Vult von Steyern P (inventor). Zirconium dioxide based prostheses. AWAPATENT AB, assignee Sweden patent. World Intellectual Property Organization patent number WO/2010/101523. 2010.
- (99) Wennerberg A, Albrektsson T. Suggested guidelines for the topographic evaluation of implant surfaces. *Int J Oral Maxillofac Implants* 2000; 15: 331-344.
- (100) Lambrechts P, Inokoishi S, van Meerbeek B, William G, Braem M, Vanherle G. Classification and potential of composite luting materials. In: Marmann WH, (editor). State of the art of the CEREC method: proceedings from the international symposium on computer restorations Berlin: Quintessence; 1991: 61-90.
- (101) Kumbuloglu O, Lassila LV, User A, Vallittu PK. Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. *Oper Dent* 2006; 31: 248-255.
- (102) Matinlinna JP, Lassila LV, Vallittu PK. Pilot evaluation of resin composite cement adhesion to zirconia using a novel silane system. *Acta Odontol Scand* 2007; 65: 44-51.
- (103) Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Effect of water aging on microtensile bond strength of dual-cured resin cements to pre-treated sintered zirconium-oxide ceramics. *Dent Mater* 2009; 25: 392-399.
- (104) Aboushelib MN, Feilzer AJ, Kleverlaan CJ. Bonding to zirconia using a new surface treatment. *J Prosthodont* 2010; 19: 340-346.
- (105) Nakayama D, Koizumi H, Komine F, Blatz MB, Tanoue N, Matsumura H. Adhesive bonding of zirconia with single-liquid acidic primers and a tri-n-butylborane initiated acrylic resin. *J Adhes Dent* 2010; 12: 305-310.
- (106) Takeuchi K, Fujishima A, Manabe A, Kuriyama S, Hotta Y, Tamaki Y, et al. Combination treatment of tribochemical treatment and phosphoric acid ester monomer of zirconia ceramics enhances the bonding durability of resin-based luting cements. *Dent Mater J* 2010; 29: 316-323.
- (107) Yun JY, Ha SR, Lee JB, Kim SH. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. *Dent Mater* 2010;26: 650-658.
- (108) Yang B, Barloi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. *Dent Mater* 2010; 26: 44-50.
- (109) Zhang S, Kocjan A, Lehmann F, Kosmac T, Kern M. Influence of contamination on resin bond strength to nano-structured alumina-coated zirconia ceramic. *Eur J Oral Sci* 2010; 118: 396-403.
- (110) Aboushelib MN. Evaluation of zirconia/resin bond strength and interface quality using a new technique. *J Adhes Dent* 2011; 13: 255-260.

- (111) Moon JE, Kim SH, Lee JB, Ha SR, Choi YS. The effect of preparation order on the crystal structure of yttria-stabilized tetragonal zirconia polycrystal and the shear bond strength of dental resin cements. *Dent Mater* 2011; 27: 651-663.
- (112) Kulunk S, Kulunk T, Ural C, Kurt M, Baba S. Effect of air abrasion particles on the bond strength of adhesive resin cement to zirconia core. *Acta Odontol Scand* 2011; 69: 88-94.
- (113) Foxton RM, Cavalcanti AN, Nakajima M, Pilecki P, Sherriff M, Melo L, et al. Durability of resin cement bond to aluminium oxide and zirconia ceramics after air abrasion and laser treatment. *J Prosthodont* 2011; 20: 84-92.
- (114) Kawai N, Shinya A, Yokoyama D, Gomi H, Shinya A. Effect of cyclic impact load on shear bond strength of zirconium dioxide ceramics. *J Adhes Dent* 2011; 13: 267-277.
- (115) Koizumi H, Nakayama D, Oba Y, Yamada K, Matsumura H. Effect of acidic primers on adhesive bonding of tri-n-butylborane initiated adhesive resin to alumina. *J Oral Sci* 2010; 52: 571-576.
- (116) Kelly JR, Benetti P, Rungruanganunt P, Bona AD. The slippery slope: critical perspectives on in vitro research methodologies. *Dent Mater* 2012; 28: 41-51.
- (117) Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. *Dent Mater* 2010; 26: e78-93.
- (118) Roeder L, Pereira PN, Yamamoto T, Ilie N, Armstrong S, Ferracane J. Spotlight on bond strength testing--unraveling the complexities. *Dent Mater* 2011; 27: 1197-1203.
- (119) Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. *J Dent* 1989; 17: 61-67.
- (120) Braga RR, Meira JB, Boaro LC, Xavier TA. Adhesion to tooth structure: a critical review of "macro" test methods. *Dent Mater* 2010; 26: e38-49.
- (121) Turp V, Sen D, Poyrazoglu E, Tuncelli B, Goller G. Influence of zirconia base and shade difference on polymerization efficiency of dual-cure resin cement. *J Prosthodont* 2011; 20: 361-365.
- (122) Beier U, Kapferer I, Dumfahrt H. Clinical long-term evaluation and failure characteristics of 1,335 all-ceramic restorations. *Int J Prosthodont* 2012; 25: 70-78.
- (123) Scherrer SS, Cattani-Lorente M, Yoon S, Karvonen L, Pokrant S, Rothbrust F, et al. Post-hot isostatic pressing: a healing treatment for process related defects and laboratory grinding damage of dental zirconia? *Dent Mater* 2013; 29: e180-90.

- (124) Phark JH, Duarte S, Jr, Kahn H, Blatz MB, Sadan A. Influence of contamination and cleaning on bond strength to modified zirconia. *Dent Mater* 2009; 25: 1541-1550.
- (125) Quaas AC, Yang B, Kern M. Panavia F 2.0 bonding to contaminated zirconia ceramic after different cleaning procedures. *Dent Mater* 2007; 23: 506-512.
- (126) Yang B, Scharnberg M, Wolfart S, Quaas AC, Ludwig K, Adelung R, et al. Influence of contamination on bonding to zirconia ceramic. *J Biomed Mater Res B Appl Biomater* 2007; 81: 283-290.
- (127) Yang B, Lange-Jansen HC, Scharnberg M, Wolfart S, Ludwig K, Adelung R, et al. Influence of saliva contamination on zirconia ceramic bonding. *Dent Mater* 2008; 24: 508-513.
- (128) Richerson D. Mechanical behavior and measurement. In: Richerson D (editor). *Modern ceramic engineering: properties, processing, and use in design*. 3rd ed. Boca Raton, FL: CRC Taylor & Francis; 2006: 211-242.
- (129) Richerson D. Final machining. In: Richerson D (editor). *Modern ceramic engineering: properties, processing, and use in design*. 3rd ed. Boca Raton, FL: CRC Taylor & Francis; 2006: 529-546.
- (130) Sato H, Yamada K, Pezzotti G, Nawa M, Ban S. Mechanical properties of dental zirconia ceramics changed with sandblasting and heat treatment. *Dent Mater J* 2008; 27: 408-414.
- (131) Kosmac T, Oblak C, Marion L. The effects on dental grinding and sandblasting on ageing and fatigue behavior of dental zirconia (Y-TZP) ceramics. *J European Ceramic Society* 2008; 28: 1085-1090.
- (132) Madani M, Chu FC, McDonald AV, Smales RJ. Effects of surface treatments on shear bond strengths between a resin cement and an alumina core. *J Prosthet Dent* 2000; 83: 644-647.
- (133) Borges GA, Sophr AM, de Goes MF, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 2003; 89: 479-488.
- (134) Kim BK, Bae HE, Shim JS, Lee KW. The influence of ceramic surface treatments on the tensile bond strength of composite resin to all-ceramic coping materials. *J Prosthet Dent* 2005; 94: 357-362.
- (135) Yamaguchi H, Ino S, Hamano N, Okada S, Teranaka T. Examination of bond strength and mechanical properties of Y-TZP zirconia ceramics with different surface modifications. *Dent Mater J* 2012; 31: 472-480.
- (136) Chintapalli RK, Marro FG, Jimenez-Pique E, Anglada M. Phase transformation and subsurface damage in 3Y-TZP after sandblasting. *Dent Mater* 2013; 29: 566-572.
- (137) Kohorst P, Borchers L, Stempel J, Stiesch M, Hassel T, Bach FW, et al. Low-temperature degradation of different zirconia ceramics for dental applications. *Acta Biomater* 2012; 8: 1213-1220.

- (138) Maerten A, Zaslansky P, Mochales C, Traykova T, Mueller WD, Fratzl P, et al. Characterizing the transformation near indents and cracks in clinically used dental yttria-stabilized zirconium oxide constructs. *Dent Mater* 2013; 29: 241-251.
- (139) Larsson C, Holm L, Lovgren N, Kokubo Y, Vult von Steyern P. Fracture strength of four-unit Y-TZP FPD cores designed with varying connector diameter. An in-vitro study. *J Oral Rehabil* 2007; 34: 702-709.
- (140) Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999; 27: 89-99.
- (141) Heikkinen TT, Matinlinna JP, Vallittu PK, Lassila LV. Long term water storage deteriorates bonding of composite resin to alumina and zirconia short communication. *Open Dent J* 2013; 7: 123-125.
- (142) Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. *Int J Prosthodont* 2002; 15: 267-272.
- (143) Kelly JR. Clinically relevant approach to failure testing of all-ceramic restorations. *J Prosthet Dent* 1999; 81: 652-661.
- (144) Mirmohammadi H, Aboushelib MN, Kleverlaan CJ, de Jager N, Feilzer AJ. The influence of rotating fatigue on the bond strength of zirconia-composite interfaces. *Dent Mater* 2010; 26: 627-633.
- (145) Anusavice KJ, Kakar K, Ferree N. Which mechanical and physical testing methods are relevant for predicting the clinical performance of ceramic-based dental prostheses? *Clin Oral Implants Res* 2007; 18 Suppl 3: 218-231.
- (146) Soderholm KJ, Geraldini S, Shen C. What do microtensile bond strength values of adhesives mean? *J Adhes Dent* 2012; 14: 307-314.
- (147) Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. *Dent Mater* 1994; 10: 236-240.
- (148) Oilo G. Bond strength testing--what does it mean? *Int Dent J* 1993; 43: 492-498.
- (149) Dunn WJ, Soderholm KJ. Comparison of shear and flexural bond strength tests versus failure modes of dentin bonding systems. *Am J Dent* 2001; 14: 297-303.
- (150) Kelly JR. Evidence-based decision making: Guide to reading the dental materials literature. *J Prosthet Dent* 2006; 95: 152-160.
- (151) Attia A, Lehmann F, Kern M. Influence of surface conditioning and cleaning methods on resin bonding to zirconia ceramic. *Dent Mater* 2011; 27: 207-213.
- (152) Yang B, Wolfart S, Scharnberg M, Ludwig K, Adelung R, Kern M. Influence of contamination on zirconia ceramic bonding. *J Dent Res* 2007; 86: 749-753.

- (153) Zitzmann NU, Galindo ML, Hagmann E, Marinello CP. Clinical evaluation of Procera AllCeram crowns in the anterior and posterior regions. *Int J Prosthodont* 2007; 20: 239-241.
- (154) Molin MK, Karlsson SL. Five-year clinical prospective evaluation of zirconia-based Denzir 3-unit FPDs. *Int J Prosthodont* 2008; 21: 223-227.
- (155) Sax C, Hammerle CH, Sailer I. 10-Year Clinical Outcomes of Fixed Dental Prostheses with Zirconia Frameworks. *Int J Comput Dent* 2011; 14: 183-202.
- (156) Sagirkaya E, Arikan S, Sadik B, Kara C, Karasoy D, Cehreli M. A randomized, prospective, open-ended clinical trial of zirconia fixed partial dentures on teeth and implants: interim results. *Int J Prosthodont* 2012; 25: 221-231.
- (157) Friederich R, Kern M. Resin bond strength to densely sintered alumina ceramic. *Int J Prosthodont* 2002; 15: 333-338.
- (158) Blatz MB, Sadan A, Soignet D, Blatz U, Mercante D, Chiche G. Long-term resin bond to densely sintered aluminum oxide ceramic. *J Esthet Restor Dent* 2003; 15: 362-8.
- (159) Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. Strength and reliability of surface treated Y-TZP dental ceramics. *J Biomed Mater Res* 2000; 53: 304-313.
- (160) Zhang Y, Lawn BR, Rekow ED, Thompson VP. Effect of sandblasting on the long-term performance of dental ceramics. *J Biomed Mater Res B Appl Biomater* 2004; 71: 381-386.
- (161) Guazzato M, Quach L, Albakry M, Swain MV. Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. *J Dent* 2005; 33: 9-18.
- (162) Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dent Mater* 1999; 15: 426-433.
- (163) de Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic. *Dent Mater* 2009; 25: 172-179.
- (164) Heintze SD, Rousson V. Survival of zirconia- and metal-supported fixed dental prostheses: a systematic review. *Int J Prosthodont* 2010; 23: 493-502.
- (165) Larsson C, Wennerberg A. The clinical success of zirconia-based crowns: a systematic review. *Int J Prosthodont* 2014; 27: 33-43.

ISBN 978-91-7104-539-3 (print)

ISBN 978-91-7104-540-9 (pdf)

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