Marginal Integrity of CAD/CAM Fixed Partial Dentures

Martin Rosentritt^a, MS
 Michael Behr^b, DDS, PhD
 Carola Kolbeck^c, DDS
 Gerhard Handel^d, DDS, MS, PhD

ABSTRACT

Objectives: Computer-aided design (CAD) and manufacturing (CAM) allows the milling of high strength zirconia fixed partial dentures (FPD), however bonding to an inert ZrO₂ ceramic surface may effect the marginal integrity of the FPDs. The aim of this investigation was to evaluate the marginal adaptation of zirconia FPDs at the interfaces between zirconia, cement, and tooth.

Methods: 32 3-unit FPDs were fabricated of the CAD/CAM Y-TZP zirconia (Lava, 3M Espe, Germany) according to the manufacturers' instructions. Resin cements with corresponding primer and bonding systems were used to lute the FPDs: Compolute/EBS multi (3M Espe, Germany), Panavia F/ED (Kuraray, Japan), Variolink 2/Syntac classic (Ivoclar-Vivadent, FL) and RelyX Unicem/without treatment (3M Espe, Germany). Au-alloy FPDs (BioPontostar, Bego, Germany) were cemented with RelyX Unicem and Harvard (Harvard, Germany) as the control. Marginal adaptation was evaluated with scanning electron microscopy using replica specimen before and after artificial aging. After aging, microleakage tests were performed with fuchsine solution. The interfaces between cement-tooth and cement-FPD were examined.

Results: At the interfaces (cement-tooth and cement-FPD), the systems showed a 95% or higher perfect margin before and after aging. Only Variolink2/Syntac had a marginal adaptation, lower than a 70% perfect margin. Generally, the fuchsine penetration was below 20%, only BioPontostar/Harvard and Lava/Variolink2 showed penetration results between 80% and 100%.

Conclusion: The success of the adhesive cementation of zirconia FPDs depends on the cement system. Under the conditions of this study, zirconia FPDs showed good to sufficient marginal integrity in combination with Panavia/ED, Compolute/EBS and RelyX Unicem. (Eur J Dent 2007;1:25-30)

Key words: CAD/CAM; Marginal integrity; Microleakage; Zirconia.

INTRODUCTION

Pressable all-ceramic materials exhibit a fracture resistance which limits the application of the materials to full coverage single unit restorations. The use of high strength hot isostatic pressed (hipped) or partly stabilized zirconia ceramics in combination with computer aided manufacturing

(CAM) represents an effort to increase the indications in which all-ceramics can be used to replace posterior teeth. The concepts extend from manufacturing "green" or "white" pre-sintered ceramic cores (Cercon, DeguDent; Lava, 3M Espe) to the milling of high strength hipped zirconia cores (Digizon, Girrbach). While all systems use CAM, modelling may follow various methodologies, from scanning a wax-up (Cercon) to computer aided design (CAD- Lava, Digizon). The veneering of the high strength cores is performed using adapted conventional layering techniques with veneering leucite-ceramics.

Conventional cementation is allowed,^{1,2} but the digitizing process often demands a conical, low retentive preparation design. Therefore resin-based bonding systems are necessary to preserve suf-

□ Department of Prosthetic Dentistry, University Clinics, Regensburg, Germany
^a Engineer
^b Assistant Professor
^c Assistant Dentist
^d Professor and Chairman
 □ Corresponding author: Martin Rosentritt
 Department of Prosthetic Dentistry,
 University of Regensburg
 D-93042 Germany
 Tel: +49 941 944 6054 Fax: +49 941 944 6171
 E-mail: martin.rosentritt@klinik.uni-regensburg.de

ficient strength of pressable and zirconia ceramics.^{3,4} To increase the surface roughness and facilitate bonding, air- abrasion with small particle Al_2O_3 , pyrosil treatment,⁵ as well as tribochemical treatments⁶⁻⁷ have been utilized, however surface treatments such as hydrofluoric etching do not help to increase the roughness of the inert zirconia surface.

Interactions at the cement-FPD transition may also influence the bonding between cement and tooth. This transition is particularly sensitive, as insufficient sealing may allow the penetration of bacteria. Under clinical conditions, the continuing decrease of marginal adaptation may culminate in a loss of the restoration due to microleakage and resulting secondary caries. Generally, dentine pre-treatment includes acid, primer, adhesive and bonding agent. It is possible to combine two or all components in one solution. The optimal adhesive cementation interlocks strongly with the hybrid layer and tag formation, but the clinical application of the materials is time consuming and technically sensitive. Cements which show comparable adaptation but avoid the complex bonding procedures are therefore desirable. New, resin based bonding materials offer the option of bonding without separate tooth treatment.^{8,9}

The aim of this investigation was to evaluate the marginal adaptation of adhesively cemented CAD/CAM-made zirconia three-unit FPDs. Additionally, new self-adhesive resin cement without surface pre-treatment was evaluated on its marginal integrity in comparison to the other bonding systems. Metal-ceramic restorations with adhesive bonding and conventional cementation were used as controls.

MATERIALS AND METHODS

The roots of 96 freshly extracted human third molars were coated with a 1 mm thick layer of polyether impression material (Impregum: ESPE, Seefeld, Germany) to simulate the resilience of the human periodontium. Two abutment teeth were embedded pair wise in PMMA resin (Palapress Vario: Kulzer, Wehrheim, Germany) at a distance of 10 mm to represent a molar gap. The circular preparation of the teeth was performed with dia-

mond burs resulting in 1 mm deep shoulders with finishing lines in dentine. Thirty-two three-unit posterior FPDs were made of the CAD-CAM System LAVA (3M Espe. Seefeld, Germany). For the fabrication of the 3-mol% yttria tetragonal zirconia polycrystal (Y-TZP), all-ceramic cores, gypsum models were optically scanned. The frameworks were designed with the computer and were milled in "green", pre-sintered ceramic condition to preserve time and milling tools. The final sintering process determines the physical properties and dimensions of the framework. The framework was veneered with the ceramic LAVA Ceram. Sixteen three unit metal FPDs (BioPontostar, Bego, Bremen, Germany) were fabricated on master casts of class IV dental stone (Fuji Rock: GC-Corporation, Tokyo, Japan) and veneered with ceramic (Omega 900; Vita, Bad Säckingen, Germany). The cements and tooth pre-treatments used are listed in the Table 1. In the case of adhesive bonding, each abutment was light cured for 2x40 sec (Elipar Trilight, 3M Espe, Seefeld, Germany).

All abutment teeth were treated depending on the bonding system, and according to the manufacturer's instructions. The inner surfaces of the FPD retainers were tribochemically treated (Rocatec Soft, 3M Espe, G, Seefeld, Germany). 24 hours after luting, excessive cement was removed with diamond burs (Meisinger, Düsseldorf, Germany) and polishing instruments (Soflex-Scheiben: 3M Dental Products, St. Paul, USA). The FPDs were then placed in an artificial oral environment for thermal cycling and mechanical loading (TCML). Thermal cycling was performed rinsing the FPDs with bi-distilled water of 5°C and 55°C, respectively. Each temperature, 5°C and 55°C was kept for two minutes. Simultaneously, the FPDs were centrally loaded with a human antagonist. A force of 50N was applied 1.2x10⁶ times with at a frequency of 1.6 Hz. Artificial aging was performed according to Krejci to simulate 5 years of intraoral service.¹⁰⁻¹¹

For the semi-quantitative analysis of the marginal adaptation, both the cement-tooth and the cement-FPD-interfaces were examined using a scanning electron microscope (SEM), Stereoscan 240 (Cambridge Instruments, Nußloch, Germany; magnification: 200x-800x; working distance 18

Table 1. Materials and manufacturers.

FPD material	Cement	Bonding	Manufacturer	Cement type
All-ceramic (LAVA/Lava Ceram, 3M Espe, G)	Variolink 2	Syntac Classic	(Ivoclar-Vivadent, FL)	Resin-cement
	Panavia F	ED	(Kuraray, J)	
	Compolute	EBS Multi	(3M Espe, G)	Universal resin- cement
	RelyX Unicem	without	(3M Espe, G)	
Metal-ceramic BioPontostar (Bego, G)/ Vita Omega (Vita, G)	RelyX Unicem	without	(3M Espe, G)	Zn-phosphate
	Harvard	without	(Harvard, G)	

mm). Replicas (Epoxy VP 1031, Vivadent, Schaan, FL, Schaan, Liechtenstein) of the cervical margins of both abutment teeth were made before and after TCML using impressions (Permadyne, 3M Espe; Seefeld, Germany) and special resin trays (diameter 50 mm). The replicas were sputtered coated with a thin layer of gold to ensure conductivity and hinder electricity charge during SEM analysis. The cervical margins, including the whole preparation line from oral over approximal to buccal were investigated and the percentage of “perfect margin” (smooth transition, no interruption of continuity) and “marginal gap” (separation of the components because of adhesive and/or cohesive failure) were determined¹² (example see. Figure 1). To improve reproducibility, one investigator evaluated all specimens. Figure 2 demonstrates the FPD design, shoulder preparation and the area, where the analysis was performed.

For the dye penetration test, the FPDs were incubated in a basic fuchsin solution (0.5%) for 16 hours at 37°C. Then the abutment teeth were cut into slices of about 500 µm thickness using a water-cooled cutter (Leica, SP1600, Bensheim, Germany). Immediately after cutting, the slices were digitized using the light microscope Stemi SV8 (Zeiss, Oberkochen, Germany) to avoid artifacts because of bleaching or water penetration.

The depth of the dye penetration (= intrusion of the fuchsin solution) and the depth of the shoulder preparation were determined using an image analysing system (Optimas 6.1, Media Cybernetics, Silver Spring, MD, USA). Percentage microleakage is defined as the percentage of dye penetration in proportion to the depth of the preparation. Figure 3 shows a photo of the dye penetration, including the principle analysis.

Median and 25/75%-percentiles were calculated. Statistical analysis was performed using the Mann-Whitney-U-Test and Kruskal-Wallis-Test for non-normal distributed values (SPSS/PC+ 5.0, SPSS, USA).¹³ The level of significance was set to $P=0.05$.

RESULTS
Marginal adaptation

At the cement-FPD interface, the cement systems showed greater than 95% perfect margins except for Lava with Variolink 2/Syntac classic, which had a significantly lower ($P=0.000$), approximately 65% perfect margin. After TCML, marginal integrity decreased for all materials by a maximum of 5% for Lava with Variolink 2/Syntac classic. The control BioPontostar/RelyX Unicem showed a perfect margin of 98% which deteriorated to 96% after TCML. BioPontostar/Harvard had values of 95%

Figure 1. Section of the cervical margin indicating the criteria for the marginal evaluation (SEM picture, x400, working distance 18mm) left: perfect margin right: marginal gap between cement and tooth.

Figure 2. FPD design with 1mm dentin shoulder preparation; arrows indicate the starting and end of the evaluation of the marginal quality.

Figure 3. Dye penetration photograph with schematic diagram of the microleakage analysis between FPD, cement and tooth.

before, and 94% after TCML (Figure 4, above).

At the cement-tooth interface, over 95% perfect margins were observed for Lava with Panavia/ED, Compolute/EBS multi and RelyX Unicem. No decrease was found after TCML. For Variolink 2/Syntac classic the integrity was 70%, which decreased significantly ($P=.042$) by 10% after TCML. BioPontostar with RelyX Unicem showed results of 97% perfect margin before and after TCML. For BioPontostar with Harvard, 95% perfect margins were found. The values decreased significantly ($P=.018$) to 93% after TCML (Figure 4, below).

The standard deviation of the marginal SEM analysis was 6.0% and the standard error 0.3%. Statistical differences were indicated in Figure 4.

Dye penetration

Lava with Panavia/ED, Compolute/EBS multi and RelyX Unicem showed penetration values lower than 5% at both cement-tooth and cement-FPD interfaces. For Variolink 2/Syntac classic, a significantly higher penetration (75%) was found at the cement-tooth interface ($P=.000$) and almost 100% ($P=.000$) at the cement-FPD interface. For the BioPontostar/RelyX Unicem FPDs, 15% leakage could be determined at the cement-tooth transi-

tion and about 6% at the cement-FPD interface. With 100% leakage at the cement-tooth transition and 0% leakage at the cement-FPD transition, the BioPontostar/Harvard FPDs showed significantly worse results (Figure 5).

The standard deviation of measurement was 28.6% and the standard error 1.5%. Statistical differences were indicated in Figure 5.

DISCUSSION

Scanning electron microscopy (SEM) evaluation is an established and reproducible procedure for determining the cervical marginal adaptation. It is based on comparable conditions and semi-quantitative analysis.¹⁴ Of course, accuracy of impression taking or replica fabrication limits the value of such procedures. The deviation of SEM analysis showed, that the semi-quantitative analysis, which was performed by one examiner, improved subjectivity and error rate. Microleakage tests with dye solution supplement the superficial marginal SEM evaluation with internal bond penetration results. The high deviations of the microleakage measurements may be a result of individual differences in tooth tissue combined with the variability of bonding quality. Microleakage and SEM results may differ, even when main parts of the circular margin are sealed ("perfect margin"): dye solution may penetrate through remaining small gaps or even the cement may chemically absorb and forward the basic fuchsin solution. Only a sealed hard tooth structure completely may hinder microleakage.

In SEM evaluation and microleakage tests, the marginal adaptation showed low penetration and a high percentage bonding between adhesive and LAVA FPDs. Only for Variolink2/Syntac classic was insufficient results found. In contrast to glass-ceramics, the inert zirconia ceramic can not be etched for pre-treating and activating the surface.

Figure 4. SEM marginal adaptation before and after aging: criterion "perfect margin [%]"; above: transition cement-FPD, below: transition cement-tooth (median, 25% / 75% percentiles, min, max); statistically significant differences are indicated by *.

Figure 5. Dye penetration [%] after artificial aging (median, 25% / 75% percentiles, min, max); identical letters/numbers identify statistically identical results.

The applied treatment with tribochemical methods allowed a successful bonding between cement and FPD, but the bond strength between zirconia and cement requires further investigations, e.g., using tensile tests.

Clinically applied resin-based bonding systems, such as Panavia/ED or Compolute/EBS,¹⁵⁻¹⁶ as well as the new system RelyX Unicem without tooth treatment, showed high marginal integrity between cement and tooth. The bonding conditions between resin cement and tooth were determined by the condition of the dentin smear layer and the etching procedure. Highest bonding results were obtained by dissolving or removing the smear layer and the formation of resin tags,¹⁷⁻¹⁸ like provided by Panavia/ED and Variolink/Syntac. RelyX Unicem without pre-treatment showed a marginal integrity comparable to the resin cements. According to the manufacturer, the resin-based RelyX Unicem is a smear layer modifying, self-adhesive, dual curing cement. It is based on methacrylated phosphoric acid esters with several cement reactions during setting. The functional groups of the modified acrylates initiate a neutralization reaction with the basic salt and the tooth apatite. In this step, water is released which may contribute to the hydrophilicity and adaptation to dentin. Without tooth pre-treatment, the smear layer remains on the tooth surface⁸⁻⁹ and hinders the creation of resin tags in the dentin tubuli. An incomplete penetration of the smear-layer with bonding agent or cement would weaken the resulting bond. An adhesive failure may be located between bonding and hybrid layer or between hybrid layer and sound dentin.¹⁹ Therefore, systems with modified smear layers often show low bond strength values,²⁰ but for RelyX Unicem a bond strength similar to the resin cements is reported.²¹ In contrast to the other resin cements, the marginal integrity for LAVA/Variolink2/Syntac was significantly lower at the transition between cement and tooth. Similar high dye penetration was found in previous investigations.⁸ The low marginal adaptation and the high amount of dye penetration, indicate a disturbed chemical reaction of the bonding system or cement. A reason may be an inadequate activation of the bonding system due to opaque, light blocking all-ceramic core.

Regarding the metal-ceramic FPD controls, the bonding with Unicem and conventional Zinc-phosphate showed comparable marginal adaptation. The high penetration results for Harvard cement at the cement-tooth interface are remarkable. Further microleakage investigations on Zinc-phosphate cement remain to be tested with alternative dye solutions, such as methanol-blue.

CONCLUSIONS

Under the conditions of this study, zirconia FPDs showed good to sufficient marginal integrity in combination with Panavia/ED, Compolute/EBS and RelyX Unicem. Restrictions had to be made with the combination Variolink2/Syntac. The adhesive bonding of zirconia restorations in combination with a tribochemical treatment²² or air abrading with small particle Al₂O₃ is a promising application, but the results are dependent on the choice of cement²³ and bonding agent. The results suggest that RelyX Unicem without tooth treatment is comparable to an adhesive cementation with regard to marginal integrity and microleakage. The bonding mechanisms of this cement should be investigated further.

REFERENCES

1. Blatz MB, Sadan A, Kern M. Resin ceramic bonding: a review of the literature. *J Prosthet Dent* 2003;89:268-274.
2. Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann H. Fracture resistance of lithium disilicate-, alumin-, and zirconia-based three-unit fixed partial dentures: a laboratory study. *Int J Prosthodont* 2001;14:231-238.
3. Jensen ME, Sheth JJ, Tolliver D. Etched-porcelain resin-bonded full-veneer crowns: in vitro fracture resistance. *Compendium* 1989;10:336-338,340-1,344-7.
4. Behr M, Rosentritt M, Mangelkramer M, Handel G. The influence of different cements on the fracture resistance and marginal adaptation of all-ceramic and fiber-reinforced crowns. *Int J Prosthodont* 2003;16:538-542.
5. Janda R, Roulet JF, Wulf M, Tiller HJ. A new adhesive technology for all-ceramics. *Dent Mater* 2003;19:567-573.
6. Bulot D, Sadan A, Burgess J.O., Bletz M.B. Bond strength of a self-adhesive universal resin cement to Lava zirconia after two surface treatments. *J Dent Res* 2003; 81: Abstr. No 578.
7. Edelhoff D, Sorensen JA. Retention of Selected Core Materials to Zirconia Posts. *Oper Dent* 2002;27:455-461.
8. Rosentritt M, Behr M, Lang R, Handel G. Influence of Cement Type on the Marginal Adaptation of All-Ceramic MOD Inlays. *Dent Mater* 2004;20:463-469.
9. Behr M, Rosentritt M, Regnet T, Lang R, Handel G. Marginal adaptation in dentin of a self-adhesive universal resin cement compared with well-tried systems. *Dent Mater* 2004; 20:191-197.
10. Krejci I, Lutz F. In-vitro test results of the evaluation of dental restoration systems. Correlation with in-vivo results. *Schweiz Monatsschr Zahnmed* 1990;100:1445-1449.
11. Rosentritt M, Leibrock A, Lang R, Scharnagl M, Behr M, Handel G: Regensburger masticator. *Materials Testing* 1997; 3:77-80.
12. Schmalz G, Federlin M, Reich E. Effect of dimension of luting space and luting composite on marginal adaptation of class II ceramic inlays. *J Prosthet Dent* 1995;73:392-399.
13. Brosius G, Brosius F. SPSS Base System and Professional Statistics, 1st edn. Bonn, Albany: International Thomson Publishing, 1995.
14. Roulet JF, Reich T, Blunck U, Noack M. Quantitative margin analysis in the scanning electron microscopy. *Scanning Microscopy* 1989;3:147-159.
15. Van-Dijken JW. Resin-modified glass ionomer cement and self-cured resin composite luted ceramic inlays. A 5-year clinical evaluation. *Dent Mater* 2003;19:670-674.
16. Felden A, Schmalz G, Hiller KA. Retrospective clinical study and survival analysis on partial ceramic crowns: results up to 7 years. *Clin Oral Invest* 2000;4:199-205.
17. Koibuchi, Yasuda N, Nakabayashi N. Bonding to dentin with

- a self-etching primer: the effect of smear layers. *Dent Mater* 2001;17:122-126.
18. Shinci MJ, Soma K, Nakabayashi N. The effect of phosphoric acid concentration on resin tag length and bond strength of a photo-cured resin to acid-etched enamel. *Dent Mater* 2000;16:324-329.
 19. Kiyomura M. Bonding strength to bovine dentin with 4-Meta/Mma-TBB resin. Long-term stability and influence of water. *J Jpn Dent Mater* 1987;6:860-872.
 20. Koibuchi H, Yasuda, N, Nakabayashi N. Bonding to dentin with a self-etching primer: the effect of smear layers. *Dent Mater* 2001;17:122-126.
 21. Irie M, Suzuki K, Windmüller B. Effect of one-day storage on marginal gap of composite inlays. *J Dent Res* 2002;81: Abstr. No. 3365.
 22. Sahafi A, Peutzfeldt A, Asmussen E, Gotfredsen K. Bond strength of resin cement to dentin and to surface-treated posts of titanium alloy, glass fiber, and zirconia. *J Adhes Dent* 2003;5:153-162.
 23. Wegner SM, Kern M. Long-term resin bond strength to zirconia ceramic. *J Adhes Dent* 2000;2:139-147.