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Lohbauer, Ulrich; Belli, Renan; Cune, Marco; Schepke, Ulf

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Fractography of clinically fractured, implant-supported dental computer-aided design and computer-aided manufacturing crowns

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Ulrich Lohbauer¹, Renan Belli¹, Marco S Cune² and Ulf Schepke²

Abstract

Today, a substantial part of the dental crown production uses computer-aided design and computer-aided manufacturing (CAD/CAM) technology. A recent step in restorative dentistry is the replacement of natural tooth structure with pre-polymerized and machined resin-based methacrylic polymers. Recently, a new CAD/CAM composite was launched for the crown indication in the load-bearing area, but the clinical reality forced the manufacturer to withdraw this specific indication. In parallel, a randomized clinical trial of CAD/CAM composite crowns luted on zirconia implant abutments revealed a high incidence of failure within the first year of service. Fractured crowns of this clinical trial were retrieved and submitted to a fractographic examination. The aim of the case series presented in this article was to identify failure reasons for a new type of CAD/CAM composite crown material (Lava Ultimate; 3M Oral Care, St. Paul, Minnesota, USA) via fractographic examinations and analytical assessment of luting surfaces and water absorption behavior. As a result, the debonding of the composite crowns from the zirconia implant abutments was identified as the central reason for failure. The adhesive interface was found the weakest link. A lack of silica at the zirconia surface certainly has compromised the bonding potential of the adhesive system from the beginning. Additionally, the hydrolytic stress released from swelling of the resin-based crown (water absorption) and transfer to the luting interface further added to the interfacial stress and most probably contributed to a great extent to the debonding failure.

Keywords

Dentistry, fractography, computer-aided design and computer-aided manufacturing composite, adhesive failure, crown restoration, lava, ultimate, hydrolytic degradation

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Introduction

A major advance in digital restorative dentistry is the recent marketing of computer-aided design/computer-aided manufacturing (CAD/CAM) polymer materials for single crown restorations.¹ CAD/CAM polymers such as Lava Ultimate (3M Oral Care) are in fact resin composites and are generally identified as highly filled methacrylic resinous materials.² An organic nanofiller technology enabled a filler loading up to 82 vol% in a three-dimensionally cross-linked polymer matrix consisting of short- and long-chain dimethacrylate monomers. In the past two decades, mechanical properties of such materials were improved competing today with silica-based glass-ceramics for the single crown indication.³ While dental composites have traditionally been applied in a direct way—via viscous paste insertion, intraoral shaping, and subsequent light

curing—the idea became appealing to further improve the mechanical performance by pre-polymerization. With the aid of pressure, temperature, a suitable initiator system, and

¹Research Laboratory for Dental Biomaterials, Dental Clinic I—Operative Dentistry and Periodontology, Friedrich-Alexander University Erlangen-Nuremberg (FAU), Erlangen, Germany

²Department of Fixed Prosthodontics, Center for Dentistry and Oral Hygiene, University Medical Center Groningen, University of Groningen, Groningen, The Netherlands

Corresponding Author:

Ulrich Lohbauer, Research Laboratory for Dental Biomaterials, Dental Clinic I—Operative Dentistry and Periodontology, Friedrich-Alexander University Erlangen-Nuremberg (FAU), Glueckstrasse 11, 91054 Erlangen, Germany.

Email: ulrich.lohbauer@fau.de



accompanied by the parallel development of high-precision CAD/CAM technologies for dentistry, CAD/CAM composites entered the market. These new CAD/CAM composite materials combine a sufficiently high flexural strength with a relative low elastic modulus.^{3,4} This is supposed to mimic the resilient periodontal ligament using a low modulus crown material and hence to prevent biomechanical complications during occlusal contact loading.⁵ The improved resilience and enamel-like wear behavior, combined with the CAD/CAM chairside treatment option made this a popular material, very competitive, and advantageous to established ceramic restoratives.

CAD/CAM composite blocks are classified as medical products which allow marketing without proof of effectiveness from extensive clinical trials. It became obvious that a variety of such materials entered the market with different promises and performances. One of the first materials on the market was Lava Ultimate indicated for all types of single-tooth restorations including the single full crown. After clinical use, the number of concerns increased in terms of fracture and debonding.^{6,7} However, the causes still remain unclear. A variety of combinations regarding support material (dentin, enamel, implant titanium or zirconia, etc.), adhesive procedure (surface pretreatment, bonding, and cementation strategy, polymerization, etc.), and material degradation (hydrolysis, mechanical fatigue and wear, etc.) are currently under discussion in dental research.⁸ One strong hypothesis, among others, guides toward a weak adhesive luting interface. Adhesion is established by a complex luting multilayer, consisting of the crown and abutment material surfaces, the thin adhesive layers, and the resin-based luting cement in between.

The aim of this study was to analyze the clinical fracture process of three Lava Ultimate crowns, bonded to zirconia implant abutments. All fractures occurred during the first year of function and within a randomized controlled clinical trial (RCT). Fractographical analysis was performed on the crown material as well as on the zirconia implant abutments in order to speculate on possible reasons for failure. Chemical surface analysis of the implant abutment surface as well as water absorption measurements of the polymeric material were performed for a deeper analytical insight into responsible mechanisms leading to failure.

Case section

The fractographic examination of three fractured clinical crowns is based on a RCT.⁹ Although the clinical procedure is described elsewhere, a brief description relevant for the fractographic examination is as follows.

The restoration material used in both treatment modalities of the RCT was a resilient (“shock-absorbing”) crown material (based on resin composites) bonded to a stiff zirconia implant abutment. A total of 50 patients with a

missing single premolar in the maxilla or mandible were included. Among other factors, severe bruxism was rated as an exclusion criterion. After implant therapy and impression taking, the abutment–crown complex was fabricated in the dental laboratory. The milled crowns were visually examined for defects by the dental technician prior to the bonding procedure. Subsequently, the zirconia abutment surfaces as well as the crown intaglio sides were sandblasted using the Rocatec system (tribochemical silica coating using Rocatec Soft (3M Oral Care), 30 μ m, 2 bar, 2–10 mm distance). The adhesive procedure made use of the 3M adhesive/cement system and was performed according to the respective instructions for use (IFU, in 2013). Scotchbond Universal (3M Oral Care) was applied on the crown intaglio as well as on the abutment surfaces (no separate light curing, as stated in the IFU). RelyX Ultimate (3M) was used as resin luting agent and light cured for 5 min in a GC Labolight device (GC Europe, Leuven, Belgium). After delivery, the crown–abutment complex was screw retained to the implant, and the access cavity was filled with a glass ionomer restoration material.

100% implant and abutment survival was evaluated, but only 14% ($n = 7$) of the abutments showed uncompromised survival after 1 year of clinical service. 80% ($n = 40$) initial debonded crowns and 6% ($n = 3$) fractured crowns were documented. In all debonding cases, the luting remnants were found in the crowns but not on the abutment side, which was not always the case for the fractured crowns. The study was the first published clinical trial on the performance of Lava Ultimate restorative material for single crowns.

The fragments of the three fractured crowns were collected and cleaned in an ultrasonic alcohol bath for 5 minutes and stored dry prior to further observation. The fragments were photographed with standardized illumination and equipment (Nikon D100, Medical-Nikkor 120 mm; Nikon, Tokyo, Japan) and observed under a stereomicroscope (SV6; Zeiss, Göttingen, Germany) using lateral illumination. The fractured crowns were then coated with gold for examination under a scanning electron microscope (SEM; Leitz ISI SR 50, Akashi, Japan). The fractographic examination was conducted using a systematic approach,¹⁰ and interpretations of the fracture patterns were based on established methods.¹¹ Arrest lines, hackle, wake hackle, compression curls, or any other characteristic features were identified in order to trace back crack origins, direction of crack propagation (DCP), and discriminatory indicators of crack initiation/acceleration mechanisms. Wear facets and the exposed fractured surfaces were cautiously examined using SEM standard and back-scattered modes. Analysis regarding the presence of silica on the surfaces of the zirconia abutments was performed using energy-dispersive X-ray spectroscopy (EDS) and Raman spectroscopy. Water sorption of the crown material was measured according to ISO 4049:2009.¹²

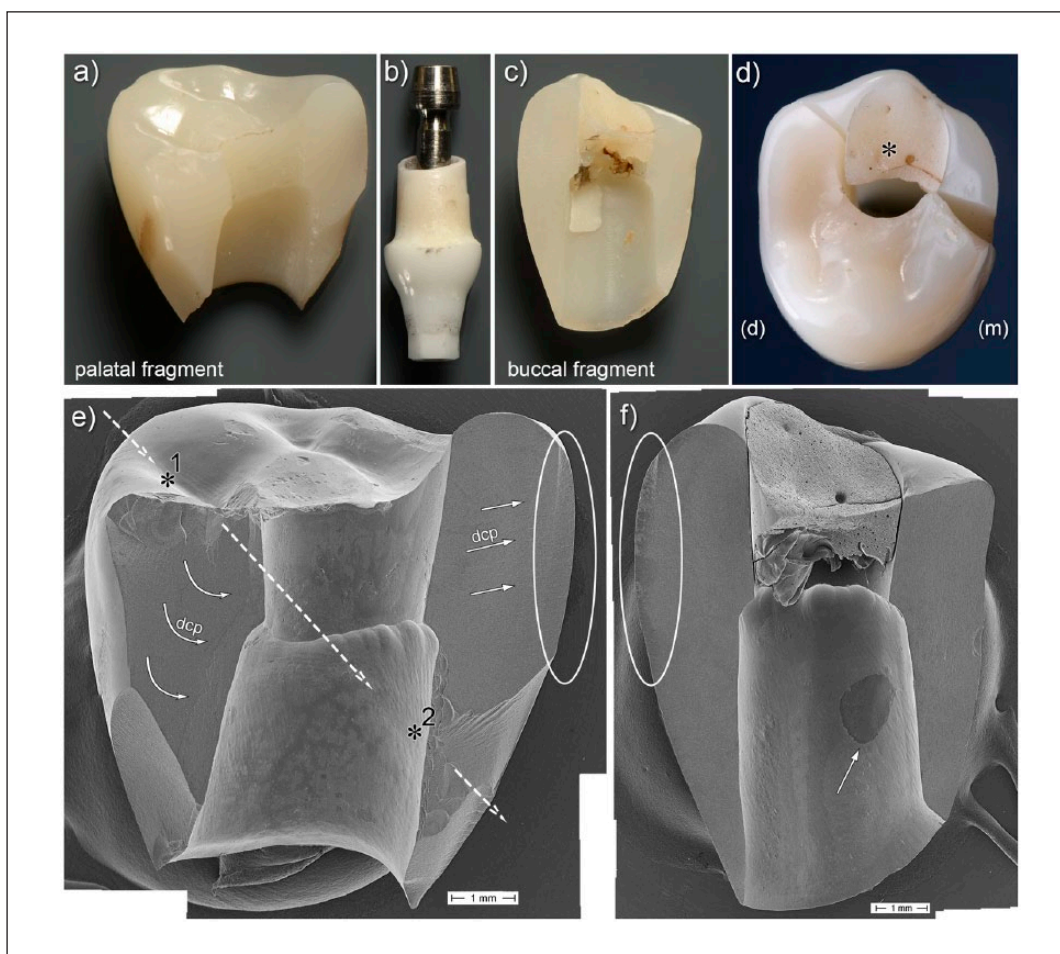


Figure 1. (a–d) Photographs of the crown and the abutment of case 1. The crown fractured mesio (m)- distally (d) in two fragments, while some minor fragments on the mesial side went missing, most likely during intraoral fracture. (b) Luting remnants can be found on the implant abutment surface. (d) The screw hole clinically filled with glass ionomer cement (*). (e and f) Higher resolution SEM images (mapped from individual SEM images) from the fracture surfaces of the fragments shown in (a) and (c). Both fragments exhibit the corresponding compression curl (circle), indicating the termination of the fracture event. (e) The palatal fragment shows clear fracture patterns on the cusp of the mesial proximal ridge (*1) and on the intaglio side of the disto-buccal margin (*2). The buccal fragment shows only minor luting remnants on the intaglio side (arrow). Most of the yellowish luting remnants are found on the zirconia abutment (b), indicating the crown–adhesive interface as the weakest link. In consequence, it seems very likely that the crown fracture was preceded by a debonding event, most likely leading to wedging of the crown and subsequent inclined shear loading in the direction assigned in (e) (dotted arrows). The fracture origin on the distal intaglio surface (*2) is thus termed a secondary event leading to abfraction of the missing marginal fragment. (e) The general direction of crack propagation (dcp) is indicated with arrows. The fracture started on the mesial side and ended on the disto-buccal side.

Case 1

This crown was retrieved from tooth #14 of a 60-year-old male patient and fractured after 2 months in situ without any complications or reported malfunction. The fractographic examination is presented in Figures 1 and 2.

Case 2

This crown was retrieved from tooth #25 of a 39-year-old female patient and fractured after 5 months in situ without any signs of malfunction. The fractographic examination is presented in Figures 3 and 4.

Case 3

This crown was retrieved from tooth #35 of a 24-year-old male patient and fractured after 4 months in situ. The patient reported that he felt loosening of the crown before mastication fracture. The fractographic examination is presented in Figures 5 and 6.

Discussion

Based on the fractographic analysis performed here, the crowns fractured mesio-distally and debonded most likely prior to the fracture event. This seems even more likely

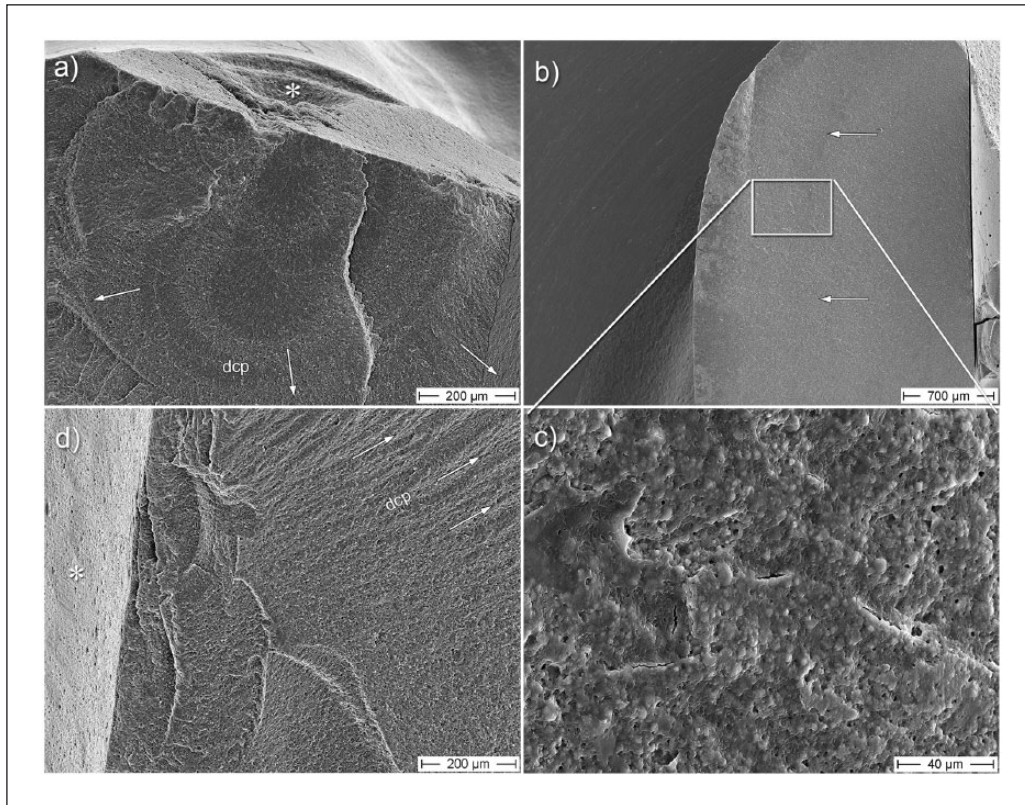


Figure 2. (a) The fracture origin on the palatal fragment of Figure 1 (e) (see *1). A large wear facet and damage accumulation zone can be seen on the surface (*) as well as multiple and overlaying mist and hackle regions and radial crack extension (arrows). (b) A magnification of the compression curl of Figure 1 (e) and (f), the endpoint of the fracture event on the disto-buccal side of the crown. (c) A high magnification of a fracture region close to the compression curl in (b). Small cracks are observable hypothesizing a degradation artifact in the microstructure of the resin composite crown material. (d) The secondary fracture event. Hackle indicates the dcp (arrows) radial from the fracture origin. (d) A clean intaglio surface (*) without any remnants of the luting agents, indicating an adhesive failure at the crown–adhesive interface.

due to the fact that 80% of the crowns debonded from the zirconia implant abutment. Interestingly, the weakest link leading to debonding was identified either at the zirconia abutment–adhesive or at the adhesive–crown interface side. A clear conclusion cannot be drawn from the three cases. However, supported by the clinical observations, the zirconia–adhesive interface seems to be more relevant for the debonding event. A cohesive fracture within the luting agent has not been observed and can thus be excluded.

The used adhesive has shown sufficient bonding performance to both the resin-based composites and to zirconia surfaces.^{13–15} Previous research has indicated that the reasons for the debonding can be manifold.¹⁶ Some of the potential causes are water of the polymeric crown material,⁸ insufficient polymerization of the adhesive under an opaque crown,¹⁷ or false marginal design or fit of the crown. The adhesion performance to zirconia and the hydrolytic changes of the resin-based crown material are relevant for this study. Based on the retrieved fragments, the zirconia abutment surface was further

chemically analyzed using Raman spectroscopy, and the of the crown composite was measured according to ISO 4049:2009.¹²

In order to establish a durable bond to zirconia surfaces, two approaches are clinically applied: chemical bonding via functional phosphate ester monomers and functionalization via tribochemical silica coating with subsequent silanization of the silica sites.^{18–20} The clinical procedure provides specific silanes for silica surfaces and specific zirconia primers containing functional monomer such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP).¹⁵ The RCT which this case study refers to, applied both approaches, that is, tribochemical coating the zirconia abutments with alumina-coated silica particles and the use of functional monomers.^{6,21} Figure 7 shows the surface analysis of the zirconia implant abutment exhibiting clear, rough patterns of an air-abraded surface but no indication of silica. This has been analyzed using SEM-EDS spectroscopy analysis. However, some alumina (remnant from sandblasting) has been found attached on the zirconia surface. Due to the lack of silica on the zirconia surface, one

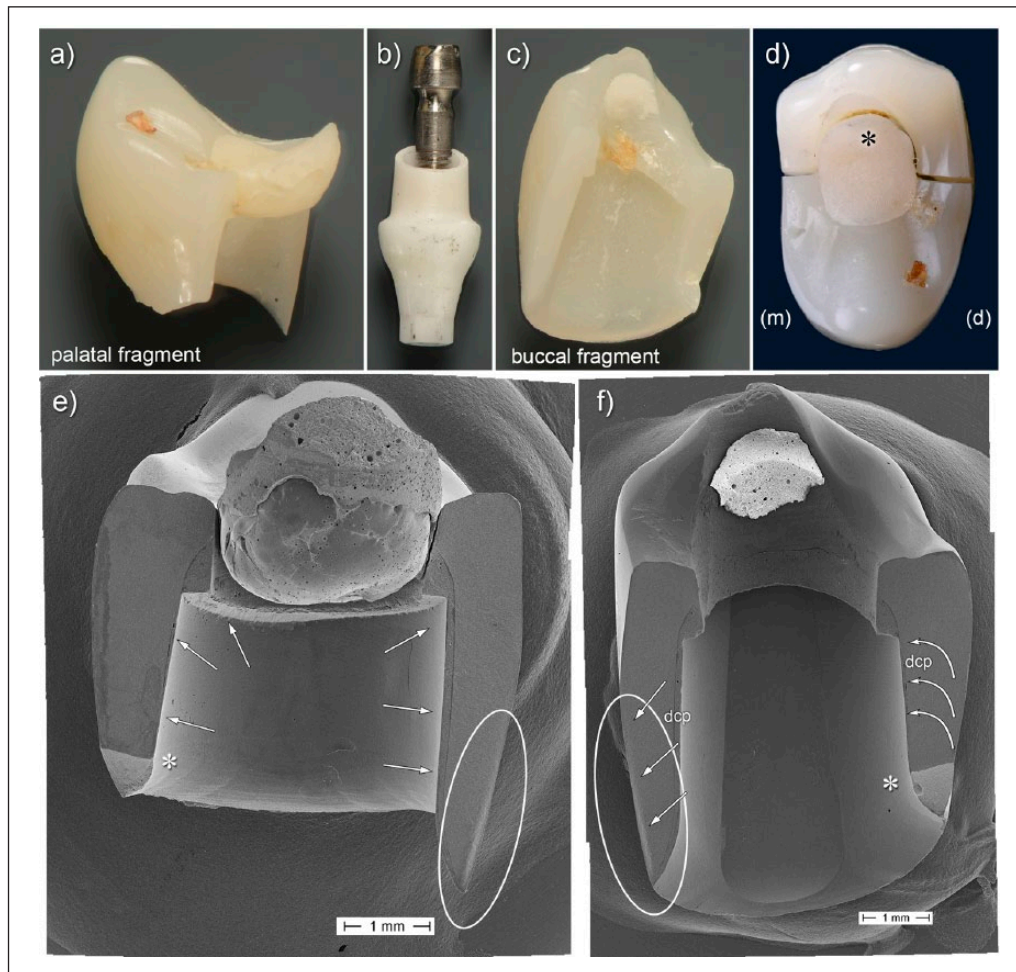


Figure 3. (a–d) Photographs of the crown and the implant abutment of case 2. The crown fractured mesio (m)-distally (d) in two fragments. (b) The zirconia abutment was found free of luting remnants. (d) The screw hole clinically filled with glass ionomer cement (*). (e) and (f) Higher resolution SEM images (mapped from individual SEM images) from the fracture surfaces of the fragments shown in (a) and (c). Both fragments exhibit the corresponding compression curl on the mesial margins (circles), indicating the termination of the fracture event. On the opposite (distal) margins, (e) and (f) shows a sharp and tilted fracture plane, suggesting the fracture initiation site at the marginal ridge (*). A clear fracture origin cannot be located, but fine hackle lines trace back to the marginal ridge, as shown in Figure 4(d). Luting remnants can be found only on the crown intaglio surface as indicated by the arrows in (e), indicating the zirconia–adhesive interface as the weakest link. It is likely that the fracture was preceded by a debonding event, resulting in tilting of the crown and shear fracture originating from the crown margins. (f) The general direction of crack propagation (dcp) is indicated by arrows. The fracture started on the distal side and terminated on the cervical mesial margin.

should conclude that adhesion is compromised. Sandblasting of tetragonal stabilized zirconia (TZP) is known to transform and degrade the microstructural cohesion of the zirconia (sub-) surface.^{22,23} Such degradation could have an effect on adhesion as well. However, the Raman spectrum in Figure 7 confirms the sole existence of the tetragonal zirconia on the buccal side of the implant abutment and no signs of the monoclinic polymorph. Apparently, no substantial tetragonal-to-monoclinic phase transformation was induced during sandblasting, which suggests that the applied sandblasting procedure (< 50 μm , 2 bar, 2–10 mm distance) was too soft. This would explain the absence of silica on the zirconia surface, as silica on

the silicatized alumina particles need impact energy to tribochemically adhere onto the zirconia surface.

The load-bearing capacity and load transfer through the whole system during mastication are also interesting to note regarding this specific crown–implant configuration. The elastic properties of the involved materials ($E_{\text{Lava Ultimate}}$: 11 GPa;⁴ $E_{\text{luting agent}}$: 6 GPa;²⁴ E_{zirconia} : 208 GPa⁴) imply that the luting composite has to bear most of the occurring stress during functional chewing. The damping behavior of the natural periodontal ligament is replaced by an osseointegrated titanium implant.^{5,24}

Adding up to this localized stress state, a resin-based methacrylic composite is known to take up water over time

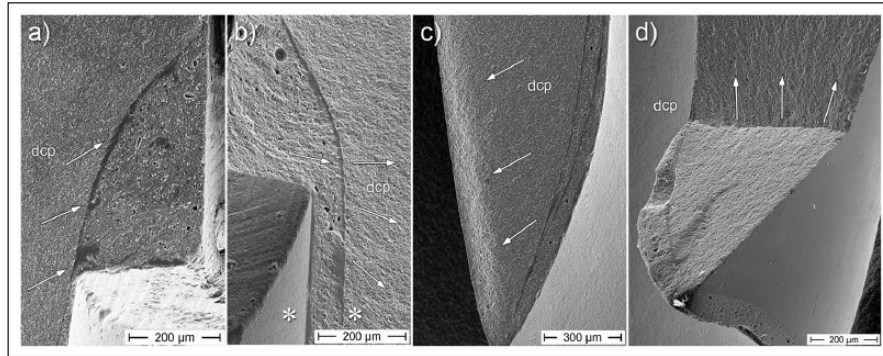


Figure 4. (a–d) High-resolution SEM images from the luting layer (Figure 3(a, b)), the compression curl (Figure 3(c)) and the fracture initiation site (Figure 3(d)). While Figure 3(a) and (b) is taken from the palatal fragment, Figure 3(c) and (d) is taken from the buccal fragment. Figure 3(a) and (b) clearly show the adhesive layer on the zirconia–adhesive side as well as on the crown–adhesive side with the sandwich luting agent in between (see * in (b)). The dcp can be seen from fine hackle lines, especially in the smooth and flat adhesive layer, indicated by arrows in Figure 3(a) and (b). Figure 3(c) and (d) also shows the luting layer and the dcp, indicated by arrows. An overview of the general dcp is shown in Figure 3(f).

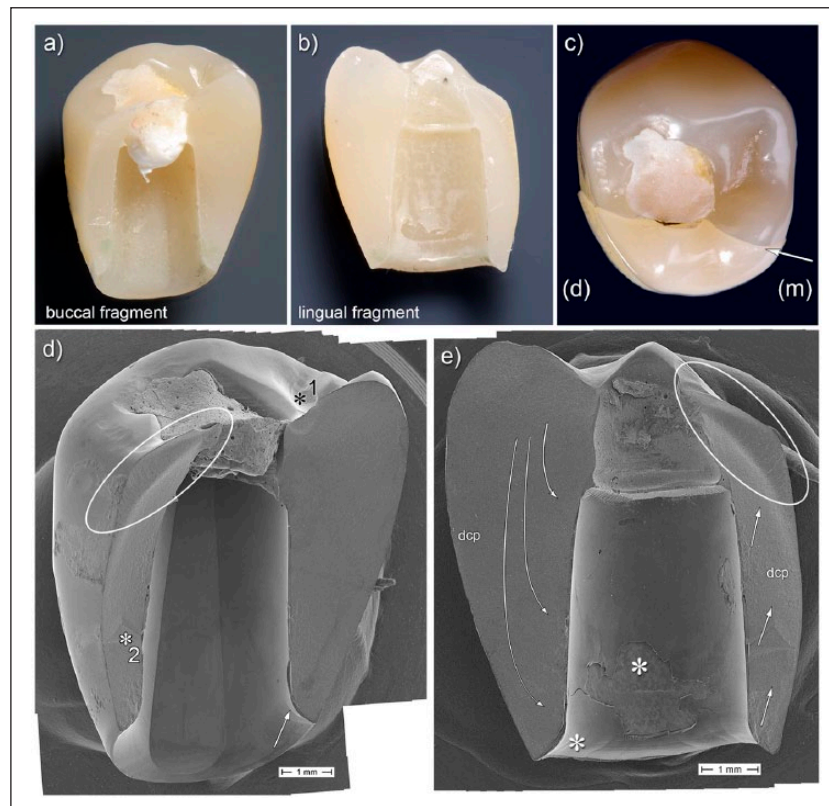


Figure 5. (a and b) Photographs of the retrieved fragments of case 3. The crown fractured mesio (m)–distally (d) in two fragments. The corresponding abutment was not available for fractographic analysis due to further patient treatment. (c) The occlusal view on the crown with the screw hole filled with glass ionomer cement (*) and the fracture plane eccentrically located on the lingual side, separating the lingual cusp (arrow). Interestingly, the fracture started on the massive and bulky lingual cusp which indicates an unbalanced masticatory loading. (d and e) Higher resolution SEM images (mapped from individual SEM images) from the fracture surfaces of the fragments shown in (a) and (b). Both fragments exhibit the corresponding compression curl on the distal side (circles), indicating the termination of the fracture event. On the opposite (mesio-lingual) side, (d) clearly shows the fracture origin (*1). A secondary fracture event is further located on the inner plane on the gingival third of the distal margin (*2). Fine hackle lines, especially in the adhesive layer (see further analysis in Figure 6), trace the general dcp as indicated in (e). The fracture started on the mesio-lingual cusp and terminated on the distal side. Luting remnants can be found on the crown intaglio surface. Some remnants broke off, as shown in (d) (arrow) and (e) (*). The luting layer tends to delaminate from either the crown or the zirconia abutment. In principle, both adhesive interfaces (but predominantly the adhesive–zirconia interface) represent the weak link leading to crown debonding.

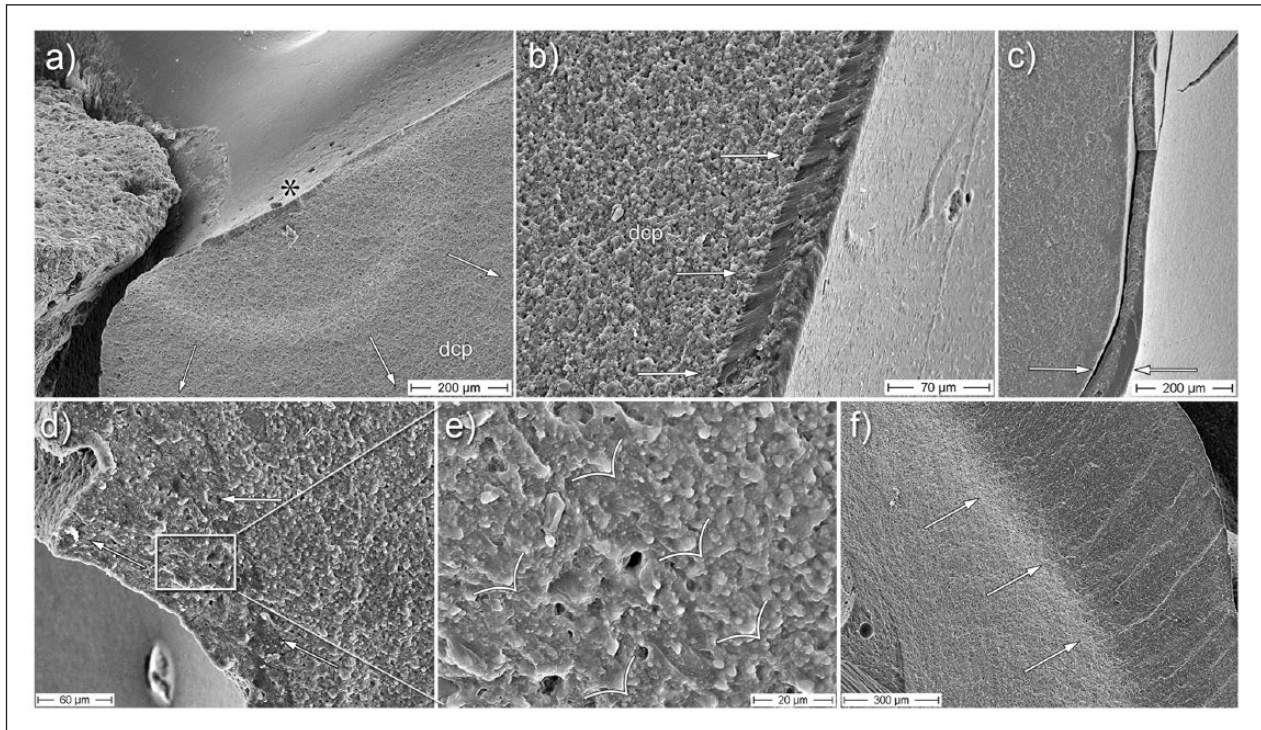


Figure 6. (a–e) High-resolution SEM images from the (a) fracture origin (b–e) luting layer, (f) the compression curl. While (a) is taken from the buccal fragment, (b–f) show magnifications of the lingal fragment. (a, b, d, f) Arrows indicate the dcp. (a) The fracture origin with a fracture releasing subsurface defect (*) and the radial fracture mirror and hackle region. The microstructure of the resin-based crown material did not elucidate distinct fractographic patterns. (b) The smooth adhesive layer, on the other hand, clearly indicates the dcp. Parallel, crazing-like hackle lines are found. (c) Delamination of the luting layer occurred mainly from the zirconia abutment, but also from the crown surface (arrows). (d and e) Also, the microstructure of the luting agent indicates the dcp. A magnification of the microstructure in (e) exhibits gull-wing-like microstructural features indicating the dcp. (f) The compression curl as the endpoint of the fracture event.

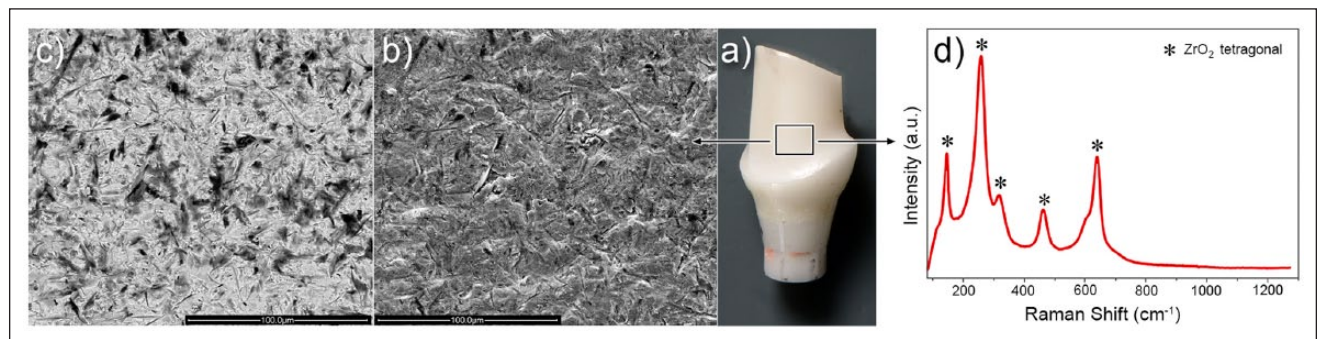


Figure 7. (a) Analytical investigations on the buccal side of one debonded zirconia implant abutment. (b) SEM surface reveals rough patterns due to sandblasting. (c) EDS analysis did not show signs of silicon in the region of interest. (d) The Raman spectrum did only indicate tetragonal zirconia and no signs of silica.

and to suffer from hydrolytic degradation and dimensional swelling to a certain extent.²⁵ Figure 8 shows a water saturation plot of the crown composite Lava Ultimate. The material takes up a maximum of $43 \mu\text{g}/\text{mm}^3$ water over 2-month saturation period. The 95% water saturation level is already reached after 14 days of water storage. The maximum of $43 \mu\text{g}/\text{mm}^3$ actually exceeds the maximum threshold value (40

$\mu\text{g}/\text{mm}^3$) in ISO 4049:2009 for polymer-based filling, restorative, and luting materials.¹² Although not measured, a relative dimensional change of a clinically placed crown can be assumed, based on the high amount of absorbed water, in turn increasing the stress state at the interface between crown and the zirconia abutment. Dental literature has not paid much attention to the swelling of resin luting agent, but indications

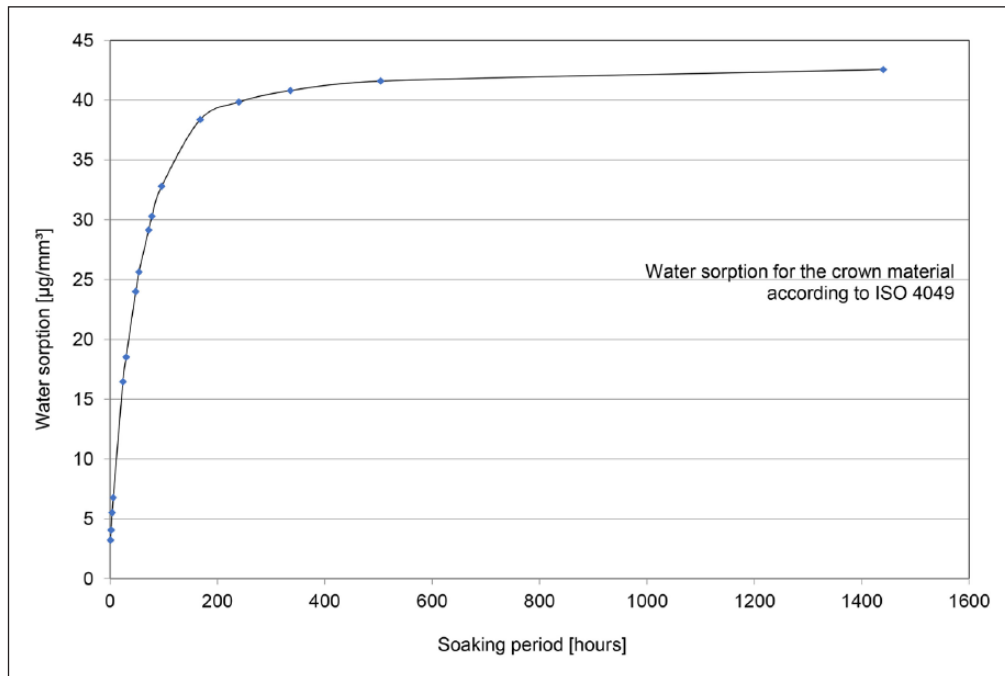


Figure 8. Water saturation curve for the resin-based composite Lava Ultimate over 2-month water storage period according to ISO 4049:2009. The material absorbed 43 $\mu\text{g}/\text{mm}^3$ water.

are available that the hydrolytic expansion stress might be responsible for crown fractures.^{26,27}

Conclusion

In combination with a preceding clinical trial, this fractographic case series underlined the debonding of the resin-based crowns from the zirconia implant abutments being the central reason for fracture. The adhesive interface was identified as the weakest link. A lack of silica at the zirconia surface certainly has compromised the bonding potential of the adhesive system from the beginning. Additionally, the hydrolytic stress induced from swelling of the resin-based crown (water absorption) and transfer to the luting interface most probably added to the interfacial stress and contributed to a great extent to the debonding failure.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Ethical approval

Ethical approval to report this case was obtained from Medical Ethics Committee of the University Medical Center Groningen (METc number 2012.388, ABR number NL 42288.042.12).

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Informed consent

Written informed consent was obtained from the patients for their anonymized information to be published in this article.

References

1. Mainjot AK, Dupont NM, Oudkerk JC, et al. From artisanal to CAD-CAM blocks: state of the art of indirect composites. *J Dent Res* 2016; 95: 487–495.
2. Chen C, Trindade FZ, de Jager N, et al. The fracture resistance of a CAD/CAM Resin Nano Ceramic (RNC) and a CAD ceramic at different thicknesses. *Dent Mater* 2014; 30: 954–962.
3. Wendler M, Belli R, Petschelt A, et al. Chairside CAD/CAM materials. Part 2: flexural strength testing. *Dent Mater* 2017; 33: 99–109.
4. Belli R, Wendler M, de Ligny D, et al. Chairside CAD/CAM materials. Part 1: measurement of elastic constants and microstructural characterization. *Dent Mater* 2017; 33: 84–98.
5. Rosentritt M, Schneider-Feyrer S, Behr M, et al. In vitro shock absorption tests on implant-supported crowns: influence of crown materials and luting agents. *Int J Oral Maxillofac Implants*. Epub ahead of print 18 May 2017. DOI: 10.11607/jomi.5463.
6. Schepke U, Meijer HJ, Vermeulen KM, et al. Clinical bonding of resin nano ceramic restorations to zirconia abutments: a case series within a randomized clinical trial. *Clin Implant Dent Relat Res* 2016; 18: 984–992.

7. Bhatia et al versus 3M Company. Lawsuit. U.S. District Court for the District of Minnesota Case number 16-CV-01304-DWF-JSM. https://www.docketalarm.com/cases/Minnesota_District_Court/0--16-cv-01304/Bhatia_et_al_v_3M_Company/
8. Flury S, Schmidt SZ, Peutzfeldt A, et al. Dentin bond strength of two resin-ceramic computer-aided design/computer-aided manufacturing (CAD/CAM) materials and five cements after six months storage. *Dent Mater J* 2016; 35: 728–735.
9. Schepke U, Meijer HJ, Kerdijk W, et al. Stock versus CAD/CAM customized zirconia implant abutments—clinical and patient-based outcomes in a randomized controlled clinical trial. *Clin Implant Dent Relat Res* 2017; 19: 74–84.
10. Scherrer SS, Lohbauer U, Della Bona A, et al. ADM guidance-ceramics: guidance to the use of fractography in failure analysis of brittle materials. *Dent Mater* 2017; 33: 599–620.
11. Quinn GD. *Fractography of ceramics and glasses: special publication 960–16*. 2nd ed. Washington, DC: National Institute of Standards and Technology, 2016, <http://nvlpubs.nist.gov/nistpubs/specialpublications/NIST.SP.960-16e2.pdf>
12. ISO 4049:2009. Dentistry—polymer-based restorative materials.
13. Keul C, Muller-Hahl M, Eichberger M, et al. Impact of different adhesives on work of adhesion between CAD/CAM polymers and resin composite cements. *J Dent* 2014; 42: 1105–1114.
14. Wagner A, Wendler M, Petschelt A, et al. Bonding performance of universal adhesives in different etching modes. *J Dent* 2014; 42: 800–807.
15. Amaral M, Belli R, Cesar PF, et al. The potential of novel primers and universal adhesives to bond to zirconia. *J Dent* 2014; 42: 90–98.
16. Rosentritt M, Hahnel S, Engelhardt F, et al. In vitro performance and fracture resistance of CAD/CAM-fabricated implant supported molar crowns. *Clin Oral Investig* 2017; 21: 1213–1219.
17. Stawarczyk B, Awad D and Ilie N. Blue-light transmittance of esthetic monolithic CAD/CAM materials with respect to their composition, thickness, and curing conditions. *Oper Dent* 2016; 41: 531–540.
18. Bomicke W, Schurz A, Krisam J, et al. Durability of resin-zirconia bonds produced using methods available in dental practice. *J Adhes Dent* 2016; 18: 17–27.
19. Bielen V, Inokoshi M, Munck JD, et al. Bonding effectiveness to differently sandblasted dental zirconia. *J Adhes Dent* 2015; 17: 235–242.
20. Inokoshi M, Poitevin A, De Munck J, et al. Bonding effectiveness to different chemically pre-treated dental zirconia. *Clin Oral Investig* 2014; 18: 1803–1812.
21. Yoshihara K, Nagaoka N, Sonoda A, et al. Effectiveness and stability of silane coupling agent incorporated in “universal” adhesives. *Dent Mater* 2016; 32: 1218–1225.
22. Grigore A, Spallek S, Petschelt A, et al. Microstructure of veneered zirconia after surface treatments: a TEM study. *Dent Mater* 2013; 29: 1098–1107.
23. Scherrer SS, Cattani-Lorente M, Vittecoq E, et al. Fatigue behavior in water of Y-TZP zirconia ceramics after abrasion with 30 μm silica-coated alumina particles. *Dent Mater* 2011; 27: e28–42.
24. Ausiello P, Ciaramella S, Fabianelli A, et al. Mechanical behavior of bulk direct composite versus block composite and lithium disilicate indirect class II restorations by CAD-FEM modeling. *Dent Mater* 2017; 33: 690–701.
25. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. *Dent Mater* 2006; 22: 211–222.
26. Sindel J, Frankenberger R, Kramer N, et al. Crack formation of all-ceramic crowns dependent on different core build-up and luting materials. *J Dent* 1999; 27: 175–181.
27. Roedel L, Bednarzig V, Belli R, et al. Self-adhesive resin cements: pH-neutralization, hydrophilicity, and hygroscopic expansion stress. *Clin Oral Investig* 2017; 21: 1735–1741.