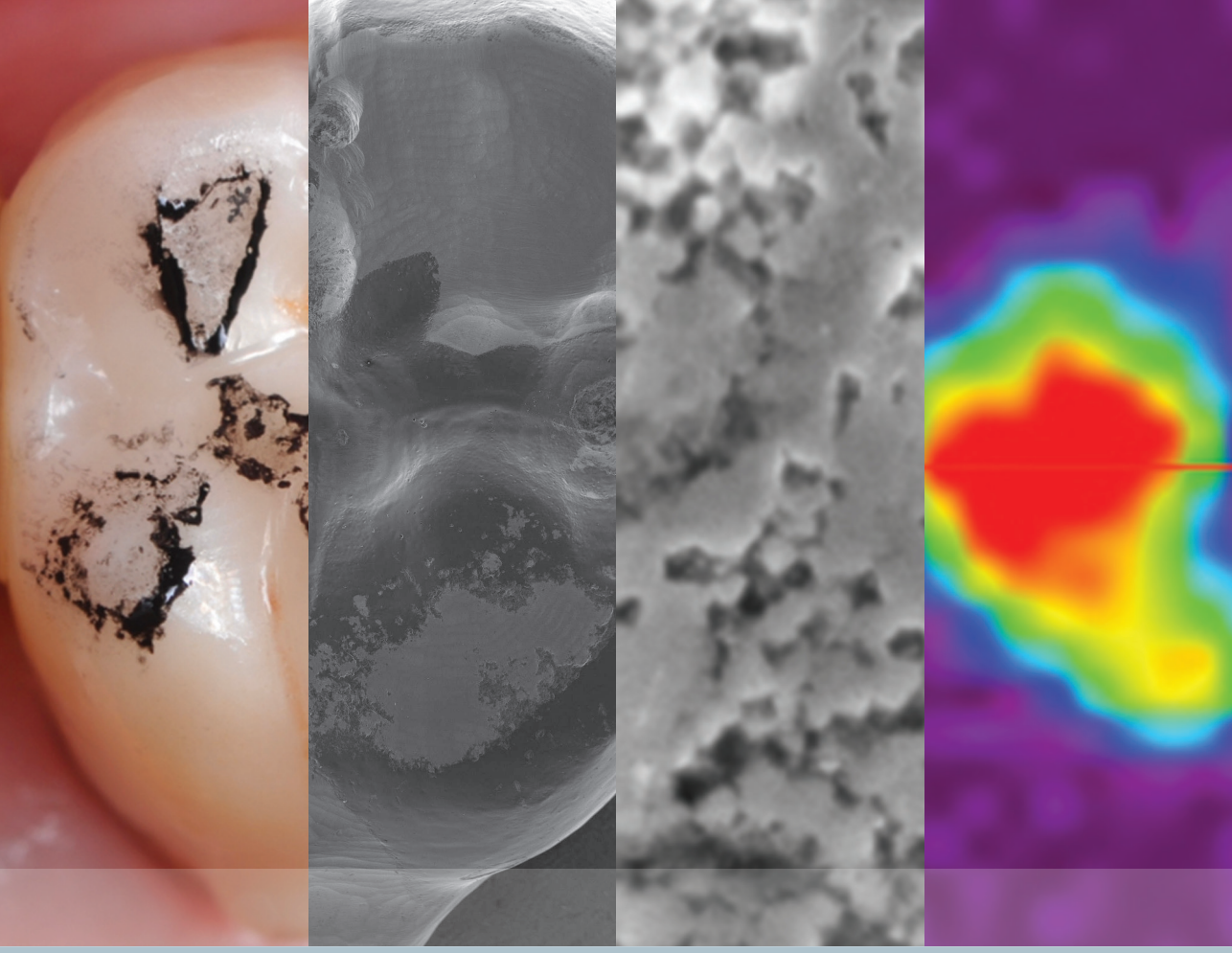


CLINICAL BEHAVIOR AND INTRAORAL LOW-TEMPERATURE DEGRADATION OF ZIRCONIA DENTAL PROSTHESES

Vinciane KOENIG

Thesis submitted to obtain the degree
of Doctor in Dental Sciences
of the University of Liege, Belgium

JURY

Promotor

Prof Amélie Mainjot, University of Liège, Belgium

Co-promotor

Prof Alain Vanheusden, University of Liège, Belgium

Jury

President : Prof Marc Lamy, University of Liège, Belgium

Secretary : Prof Rudi Cloots, University of Liège, Belgium

Members : Prof Jérôme Chevalier, University of Lyon, France

Dr Hélène Fron, University of Paris, France

Prof Julian Leprince, Catholic University of Louvain, Belgium

Dr Michaël Sadoun, Majeb company, Belgium

Prof Claudine Wulfman, University of Paris, France

ABSTRACT

The use of zirconia in the field of dental prostheses has grown significantly since its introduction in the 2000s, following the advent of computer-aided design and manufacturing technologies. First-generation zirconia-based restorations (ZBR) are bilayered structures composed of a framework and a glass-ceramic veneer, which imparts an essential esthetic appearance. However, the first clinical reports regarding veneered ZBR indicated a high rate of short-term failures due to cohesive fractures (chipping) of the veneering ceramic. To address this, a retrospective study on veneered ZBR was conducted to investigate the influence of clinical parameters, such as patient-related risk factors, on chipping failures.

Zirconia is now used to fabricate monolithic dental prostheses without the veneering ceramic layer and the only presence of a thin cosmetic glaze. These prostheses were notably developed to remedy chipping and, thus, obtain prostheses with an increased durability. Therefore, zirconia materials exhibiting greater translucency have been developed for monolithic restorations. However, second-generation zirconia materials are prone to show greater metastable behavior, which could promote low-temperature degradation (LTD).

The main objective of this study was to evaluate the intraoral LTD of zirconia monolithic restorations and the influence of occlusal stresses and glaze protection on this process. Secondary objectives included the investigation of the general clinical behavior and material wear of the restorations. This work introduces an original protocol, including *ex vivo* analyses, to evaluate the LTD process of monolithic zirconia prostheses in the oral environment and to study their general clinical behavior, primarily in terms of material wear.

LTD was shown to develop in 3 mol% yttria-doped tetragonal zirconia polycrystal monolithic restorations six months after intraoral placement and then progresses over time. After two years, the tetragonal-monoclinic transformation became non-uniform, with the presence of localized clusters of transformed grains. In axial areas, the grain aspect was typical of the classical nucleation-growth process

reported for LTD. However, in occlusal areas, tribological stress generated surface crushing and grain pull-out from the clusters, causing an underestimation of the aging degree when the evaluation was limited to monoclinic phase quantification. Glazing cannot be considered a protection against LTD because it is worn away in occlusal areas after one year.

Two years after their implantation, the Kaplan-Meier survival rate of restorations was 93.3% (100% for fixed partial dentures) and the success rate was 81.8%. It was found that eighty percent of major failures occurred in patients exhibiting clinical signs of bruxism. Complications such as root fracture, periodontal disease or composite chipping were also observed in antagonistic teeth. Wear in zirconia was observed to be less than 15 µm. The success rate of single-unit restorations was not as high as expected, the weak link being the prosthesis support or the antagonist tooth.

One hypothesis is that zirconia's stiffness and lack of ability to deform do not promote occlusal stress accommodation, which can be critical in patients affected by bruxism. Furthermore, several unexpected clinical failures were observed, including chipping and major fracture of the material, which may have been induced through LTD. If LTD occurs through the same mechanisms in dental prostheses as in orthopedic prostheses, its clinical impact remains unknown and needs to be evaluated through a thorough analysis of fractured prostheses in the framework of long-term studies.

Keywords

Computer-aided design/computer-aided manufacturing, Raman spectroscopy, *t-m* transformation, Aging, Biomaterial, Dental prosthesis, Dental implant, Wear measurement, Ceramic, Fracture, Clinical study, *Ex vivo* analysis.

RÉSUMÉ

L'utilisation de la zircone connaît un essor croissant en prothèse dentaire depuis son introduction dans les années 2000, suite à l'avènement des technologies de conception et de fabrication assistées par ordinateur (CFAO). Les restaurations à base de zircone de première génération sont des structures bicouches composées d'une infrastructure et d'une couche d' émaillage en vitrocéramique, qui en assure le rendu esthétique. Cependant, les premières études cliniques concernant les restaurations en zircone émaillées ont rapporté un taux élevé d'échecs à court terme, dus à des fractures cohésives (chipping) de la céramique d'émaillage. Pour contribuer à la compréhension de ce problème, une étude rétrospective sur les restaurations en zircone émaillées a été menée pour investiguer l'influence des paramètres cliniques, particulièrement les facteurs de risque liés au patient, sur le chipping.

La zircone est maintenant utilisée pour fabriquer des prothèses dentaires monolithiques, c'est-à-dire qu'elles ne sont pas recouvertes de céramique d'émaillage mais uniquement d'une fine couche de glaçure. Elles ont notamment été développées pour remédier au chipping et augmenter la longévité des prothèses. Dans l'objectif de réaliser ces restaurations monolithiques, des zircones spécifiques plus translucides ont été développées. Cependant, ces zircones de seconde génération sont suspectées d'avoir un comportement métastable accentué, ce qui pourrait favoriser la fatigue hydrique (low-temperature degradation, LTD).

L'objectif principal de ce travail a été d'évaluer la LTD de restaurations monolithiques en zircone en milieu buccal, ainsi que l'influence des contraintes masticatoires et de l'effet protecteur de la glaçure sur ce processus. Les objectifs secondaires ont été d'évaluer le comportement clinique général des restaurations ainsi que l'usure du matériau. Ce travail introduit un protocole original incluant des analyses *ex vivo* pour évaluer le processus de la LTD au niveau des prothèses monolithiques en zircone dans l'environnement oral ainsi que pour étudier leur comportement clinique général, notamment en terme d'usure du matériau.

Les résultats ont montré que la LTD se développe au niveau des restaurations monolithiques en zircone 3Y-TZP (3 mol% yttria-doped tetragonal zirconia polycrystal) 6 mois après leur placement en bouche et que ce processus augmente avec le temps. Après 2 ans, la transformation tétragonale-monoclinique était non-uniforme, avec la présence de clusters de grains transformés. Dans les zones axiales, l'aspect des grains était typique du processus de germination-croissance déjà rapporté pour la LTD. Cependant, dans les zones occlusales, les contraintes tribologiques ont provoqué un écrasement et un arrachage des grains transformés à la surface du matériau, ce qui induit une sous-estimation du degré de vieillissement quand l'évaluation est limitée à la quantification de la phase monoclinique.

D'autre part, la glaçure ne peut pas être considérée comme une protection contre la LTD puisqu'elle était usée au niveau de tous les points de contact occlusaux après un an.

Deux ans après leur placement, les taux de survie et de succès des restaurations suivant la méthode de Kaplan-Meier était de 93,3 % (100 % pour les ponts) et de 81,8 %, respectivement. Quatre-vingts pour cent des échecs majeurs sont survenus chez des patients présentant des signes cliniques de bruxisme. Des complications telles que des fractures radiculaires, des pathologies parodontales ou des fractures d'obturations en composite ont également été observées au niveau des dents antagonistes. L'usure de la zircone était inférieure à 15 µm. Le taux de succès des restaurations unitaires n'a pas été aussi élevé que prévu, le maillon faible étant la dent ou l'implant supportant la prothèse, voire la dent antagoniste.

Une hypothèse est que la rigidité et le manque de capacité à se déformer de la zircone ne favorisent pas l'adaptation aux contraintes occlusales, ce qui peut être critique chez les patients bruxeurs. D'autre part, certains échecs cliniques n'étaient pas attendus, tels que la présence de chipping ou de fracture majeure du matériau, qui auraient été induits par la LTD. Si la LTD survient dans les

prothèses dentaires selon le même mécanisme que dans les prothèses orthopédiques, son impact clinique reste inconnu et doit être évalué à travers une analyse approfondie des prothèses fracturées dans le cadre d'études à long terme

Mots-clés

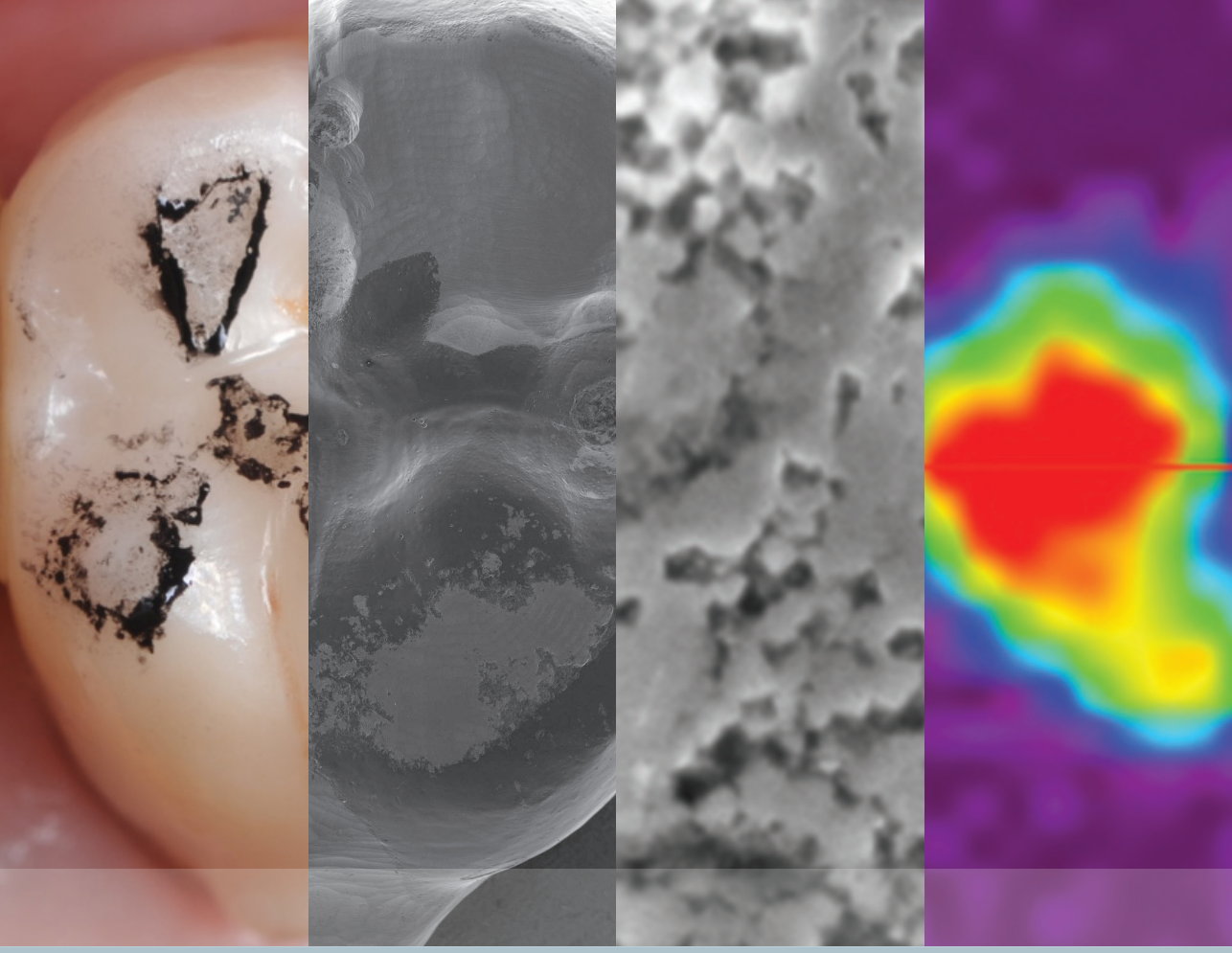
Conception et fabrication assistées par ordinateur, Spectroscopie Raman, Transformation tétragonale-monoclinique, Vieillissement hydrique, Biomatériaux, Prothèse dentaire, Implant dentaire, Quantification de l'usure, Céramique, Fracture, Étude clinique, Analyse *ex vivo*.

GLOSSARY

This glossary presents the different scientific expressions used throughout the manuscript and their abbreviations. Scientific expressions could change from one chapter to another due to journals' requirements; however, they carry the same meaning.

AFI: Accordion fringe interferometry
BOP: Bleeding on probing
***c*:** Cubic
CAD-CAM: Computer-aided design and computer-aided manufacturing
CFOA: Contact-free occlusal areas
CTE: Coefficient of thermal expansion
DP: Digital profilometry
FDPs: Fixed dental prostheses
FPDs: Fixed partial dentures
FZ: Full zirconia
GCC: Glazed centric cusp
GLMM: Generalized linear mixed model
GNCC: Glazed non-centric cusp
HIP: Hot isostatic pressed; Hot isostatic pressure
IQR: Interquartile range
LTD: Low-temperature degradation
***m*:** Monoclinic
MP: Measurement point
MPs+: Transformation-positive measurement points
MS: Matching software
OCA: Occlusal contact areas
ODP: Optical digital profilometry
PAL: Probing attachment level

PCR: Plaque control record
PFM: Porcelain-fused-to-metal
PICN: Polymer-infiltrated ceramic network
PPD: Probing pocket depth
RMS: Root mean square
SD: Standard deviation
SEM: Scanning electron microscopy
t: Tetragonal
UCC: Unglazed centric cusp
UNCC: Unglazed non-centric cusp
USPHS: United States public health service
VAS: Visual analogue scale
V_{fm}: Monoclinic volume fraction
VPSE: Variable-pressure secondary electron
XRD: X-ray diffraction
Y-PSZ: Yttria-partially stabilized zirconia
Y-TZP: Yttria-doped tetragonal zirconia polycrystal ; Yttria-tetragonal zirconia polycrystal
ZBR: Zirconia-based restorations



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Layout and graphics : perspectives.be

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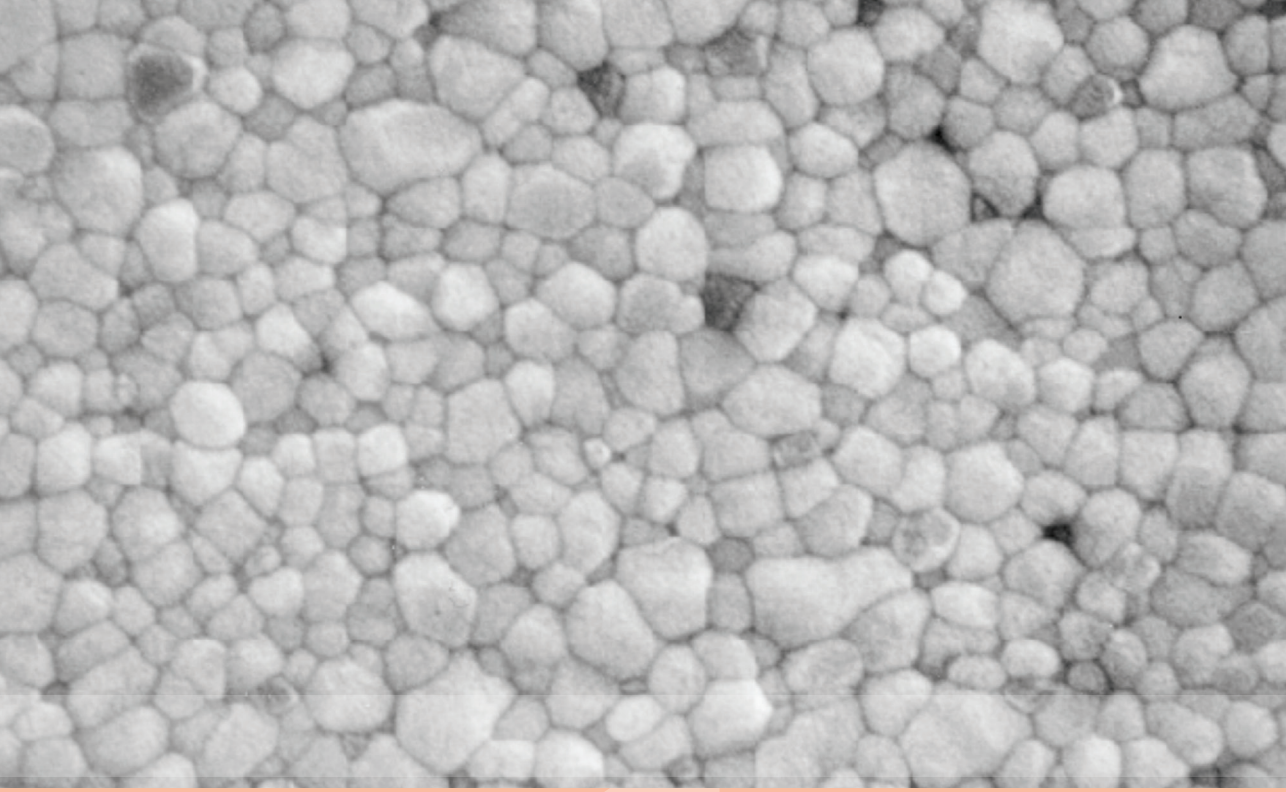

1 Introduction

1. WHITE METAL IN PROSTHETIC DENTISTRY

Computer-aided-design and manufacturing (CAD-CAM) processes revolutionized the field of dental prostheses at the end of the last millennium. The gradual disappearance of traditional processes is an economical trend offering several advantages such as manufacturing reproducibility and, more significantly, the opportunity to use high-performance materials, particularly high-strength polycrystalline ceramics and, more recently, innovative composites. The first polycrystalline ceramic to be applied in prosthetic dentistry was alumina, which was first introduced in the 1990s; however, it was progressively replaced by zirconia since the early 2000s. Zirconia materials have been well-known in the biomedical field since the 3 mol% yttria-doped tetragonal zirconia polycrystal (3Y-TZP) was first employed in the late 1980s for orthopedic prostheses (Christel 1989). In dentistry, it has been used since the 1990s as endo-osseous implants and implant abutments; however, since the 2000s, it has been used most extensively in crowns (Figure 1.1) and fixed partial dentures (FPDs, also called bridges, which are used to replace missing teeth and are single-piece multiple unit restorations composed of several crowns attached in sequence) on natural teeth and dental implants.

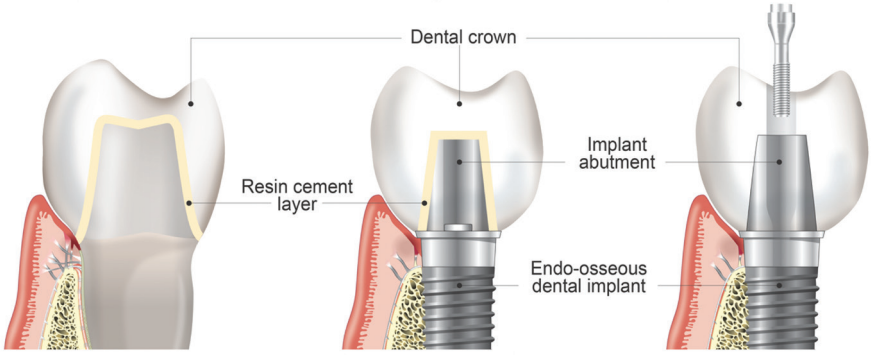

Figure 1.1 *Schematic illustration of a cemented crown on natural tooth, cemented crown on implant abutment, and screw-retained crown on implant.*

Zirconia-based dental prostheses are being used more frequently, and their use has now exceeded that of porcelain-fused-to-metal (PFM) prostheses in the United States for several years, which were the gold standard for several decades (Makhija et al. 2016). Owing to its unique phase transformation at room temperature (see section 2.1), 3Y-TZP exhibits higher strength than glass-ceramics, which are limited to be used in single crowns or small anterior bridges (Figure 1.2), and better optical properties than metal alloys; manufacturers referring sometimes to 3Y-TZP as "white metal".

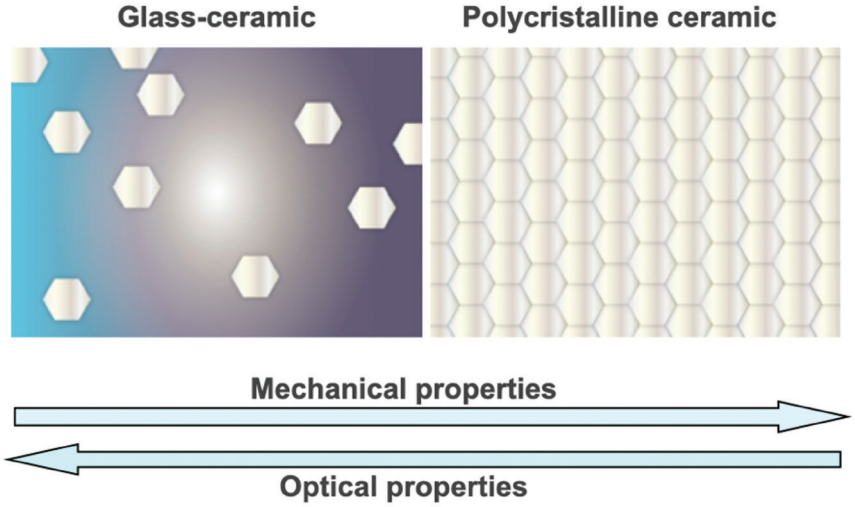

Figure 1.2 *Classification of ceramics according to microstructure (adapted from "Esthetics in dentistry," Mainjot A, in Quintessence, Edited by Devorah Schwartz-Arad, 2016). Glass-ceramics present a glass matrix containing a fraction of crystals, whereas polycrystalline ceramics contain only crystals. Crystals promote the mechanical properties of the material, whereas glass promotes its translucency and adhesion to resin composite cements. According to manufacturers, zirconia is the only dental ceramic that can be used for bridges with more than three elements. The mechanical properties of Y-TZP are directly related to its crystallography, with the evolution from a tetragonal to monoclinic phase transformation being responsible for both the strength and toughness of zirconia.*

Zirconia also has a major advantage because of its biocompatibility. *In vitro* studies have reported the strong performance of gingival fibroblasts and keratinocytes in terms of cell viability, attachment, and proliferation when

grown on Y-TZP (Grenade et al. 2016; Grenade et al. 2017; Kim et al. 2015; Moon et al. 2013; Neunzehn et al. 2012; Okabe et al. 2016). The biocompatibility properties of zirconia are similar to those of titanium and are superior to those of glass-ceramics (Grenade et al. 2016; Grenade et al. 2017); this is particularly important in implant-supported prostheses, which are in direct contact with the gingival tissue and can extend to the bone level (Figure 1.3). The so-called "biological width" (Abrahamsson et al. 1998; Abrahamsson et al. 1997), which creates a barrier to protect peri-implant structures from bacterial penetration and prevents bone resorption and gingival recession (Rompen 2012), is located immediately above the bone level and is composed of two distinct parts: a connective tissue attachment and, above it, an epithelial tissue attachment (junctional epithelium). When the implant neck is buried within the bone ("bone-level" implant type), the biological width is located at

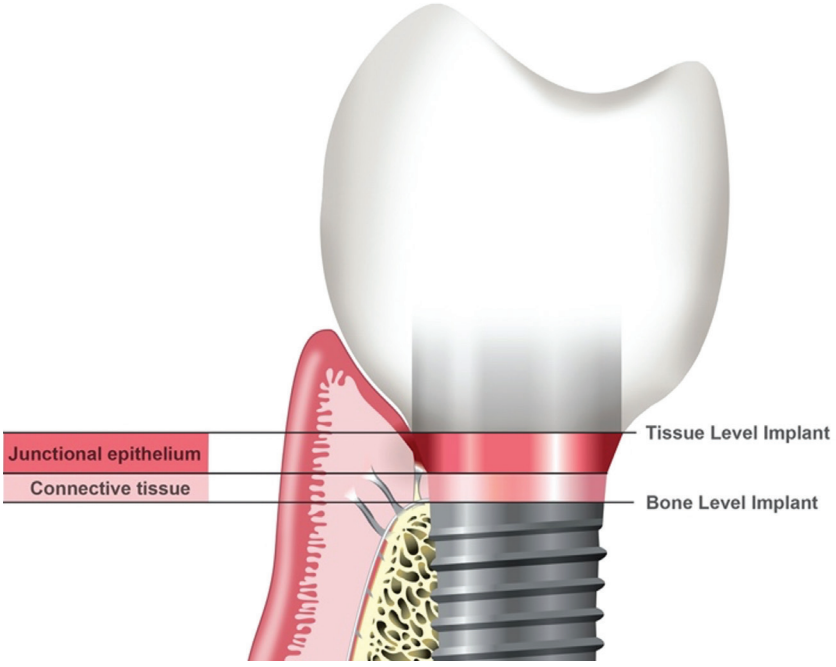

Figure 1.3 *Schematic illustration of the hard and soft tissues around an implant. The biologic width is located above the bone level, i.e., on the neck of the implant in the case of «tissue-level» implants and on the prosthesis in the case of «bone-level» implants (Grenade et al. 2016).*

the transgingival part of the abutment or screw-retained restoration, which must promote the attachment of fibroblasts and keratinocytes. Therefore, zirconia is an ideal biomaterial for abutments or screw-retained restorations. Moreover, it is not susceptible to galvanic corrosion, and its white does not affect the color of gingival tissue (Linkevicius and Vaitelis 2015), which constitutes a major advantage over titanium.

Zirconia crowns and FPDs are bilayered structures composed of a framework, which provides mechanical support for the restoration, and a hand-made glass-ceramic veneer layer, which is more brittle and ensures its esthetic appearance. However, the first clinical reports regarding veneered zirconia-based crowns and FPDs indicated a high rate of short-term failures compared to PFMs. These were a result of cohesive fractures (chipping) of the veneering ceramic (Figure 1.4), constituting the weak link of the restoration (Limones et al. 2020; Pjetursson et al. 2018; Sailer et al. 2018). Many *in vitro*

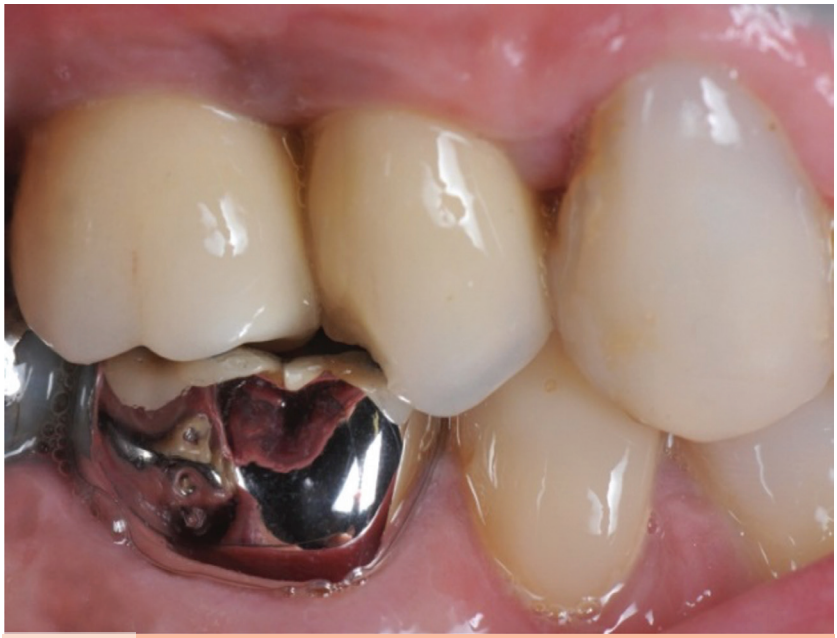

Figure 1.4 *Chipping of the veneering ceramic on a veneered zirconia-based crown (tooth #15).*

studies have been conducted in an attempt to understand and mitigate this problem, notably by investigating the influence of the manufacturing process and residual stresses (Inokoshi et al. 2016; Mainjot et al. 2015; Mainjot et al. 2011; 2012a; 2012b; Silva et al. 2011). Changes pertaining to the production of the prostheses, such as changes in the framework design, could explain the reduction in chipping failures observed during clinical studies on zirconia crowns (Sailer et al. 2018). However, it must be noted that high-risk patients, that is, those affected by bruxism (who grind their teeth and, therefore, apply significant stress on prostheses) are often excluded from clinical studies, which could significantly influence the results. This problem still persists in the long-term application of FPDs (Sailer et al. 2018).

The persistent chipping problem led to the development of monolithic restorations in the 2010s. Monolithic restorations are not covered with a glass-ceramic veneer layer and are composed entirely of zirconia. Recent advancements in dental zirconia include the development of materials exhibiting higher translucency as the high anisotropy of the refractive index (birefringence) and intrinsic opacity of first-generation Y-TZP did not impart the required esthetics for monolithic prostheses (Zhang and Lawn 2018) (see section 3). The resulting improvements in the optical properties facilitate the fabrication of restorations, which can simply be polished, stained, or glazed with a thin film of glass-ceramic (Figure 1.5). This concept improves the mechanical durability of the prosthesis, simplifies their manufacturing,

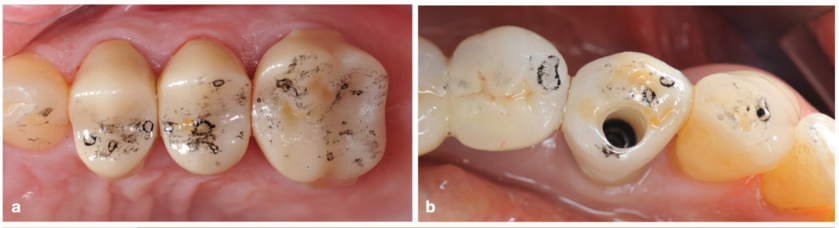

Figure 1.5 *Clinical images.*
a) Cemented crowns on teeth #24, #25, and #26 (occlusal view).
b) Screw-retained crown on tooth #34 (occlusal view). Dental lab: laboratoire Andriessens, Laurent Chiampo, Belgium.

and decreases production costs. Nowadays, restorations can be produced through CAD-CAM directly in the dental office (known as chairside restorations), using specialized equipment and without requiring the presence of a dental technician (Malkondu et al. 2016). Zirconia materials have penetrated the dental market and will play a significant role in the future of prosthodontic materials.

2. TETRAGONAL-MONOCLINIC (*T- M*) TRANSFORMATION: LIGHT AND DARK SIDES

2.1 Transformation toughening

There are three temperature-dependent crystallographic configurations of pure zirconium oxide (ZrO_2): monoclinic (*m*) (from room temperature to 1170 °C), tetragonal (*t*) (from 1170 °C to 2370 °C), and cubic (*c*) (from 2370 °C to 2680 °C, the melting point) (Figure 1.6). The addition of a dopant, particularly yttrium oxide, tend to stabilize the tetragonal structure. The resulting product, known as Y-TZP, is metastable at room temperature and can transform from *t* to a stable *m* configuration under an applied stress (Figure 1.6).

When a crack starts to propagate in the material, the tensile stresses, which are concentrated at the crack tip, trigger the *t-m* transformation locally. This is characterized by a subsequent volume increase of approximately 4% in the crystals. This expansion induces the development of compressive stresses that closes the crack, preventing its propagation (Figure 1.7). Therefore, zirconia demonstrates the highest fracture toughness among all dental ceramic materials (6–10 $MPa/m^{1/2}$) and has been advertised by manufacturers to exhibit "crack healing properties". Transformation toughening in zirconia ceramics, first described in 1975 by Garvie et al.,

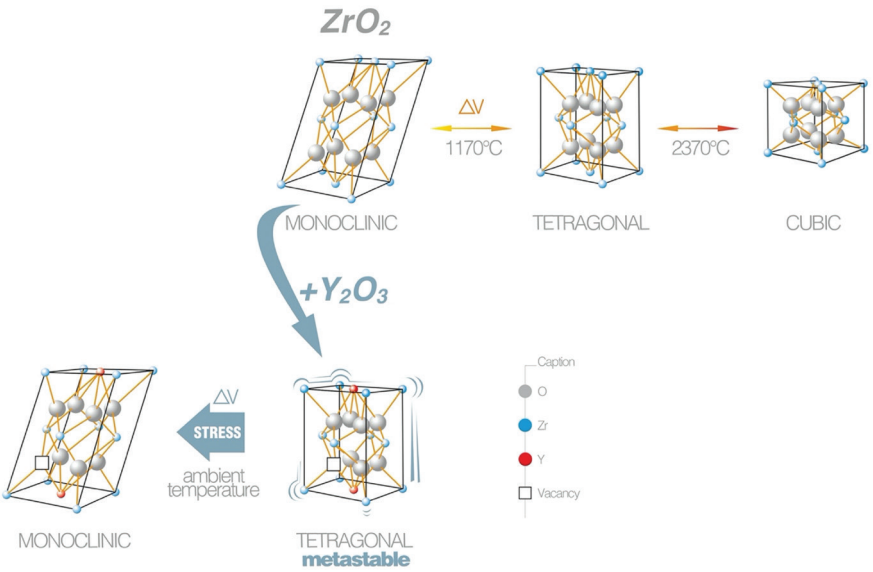

Figure 1.6 *Schematic illustration of the allotropic varieties of zirconia and tetragonal to monoclinic transformation ("Zircone(s) Partie 1 - A la rencontre de céramiques pas comme les autres ", Mainjot A, in Biomatériaux Cliniques, Vol 3, n°1, mars 2018).*

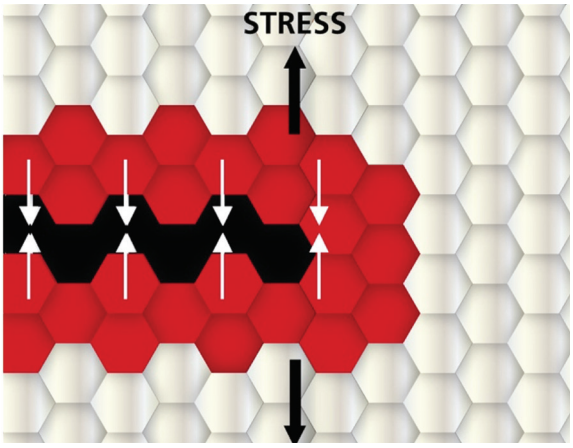

Figure 1.7 *Schematic illustration of the transformation toughening mechanism, adapted from "Zirconia ceramics and zirconia dispersed composites" Chevalier J, Gremillard L, in Bioceramics and Their Clinical Applications, Edited by T. Kokubo C R C, 2008 (from Mainjot A. Thesis, 2011)*

results in exceptional mechanical properties (Garvie et al. 1975). Thus, in addition to biomedical prostheses, zirconia ceramics have been applied in numerous industrial applications, including cutting instruments.

2.2. Low-temperature degradation (LTD)

Although the *t-m* phase transformation in zirconia materials is generally considered to be advantageous, it can also have detrimental effects. LTD in zirconia was first described in 1981 (Kobayashi 1981). It occurs through a slow surface transformation to the stable monoclinic phase in the presence of water within a certain temperature range, typically between room temperature and approximately 400 °C. Transformation begins within isolated grains at the surface and then propagates internally through a slow nucleation and growth process (Chevalier et al. 2009). Nucleation of a surface grain in contact with water induces a volume increase, which, in turn, induces stresses within the neighboring grains, triggering their phase transformation. This process results in the formation of microcracks at the surface of the material. This provides a pathway for water ingress through capillarity, initiating the process in the underlying layers (Chevalier et al. 2009) (Figure 1.8).

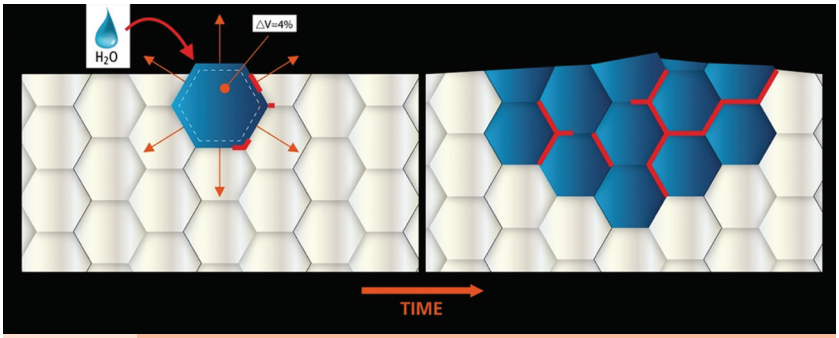

Figure 1.8 *Schematic illustration of low-temperature degradation, adapted from "Zirconia ceramics and zirconia dispersed composites" Chevalier J, Gremillard L., in Bioceramics and Their Clinical Applications, Edited by T. Kokubo C R C, 2008 (from Mainjot A. Thesis, 2011).*

Several hypotheses have been proposed to explain the role of water in the *t-m* transformation. The explanation that is most frequently provided in the literature describes the initiation of this transformation as the filling of oxygen vacancies (Chevalier et al. 2009). The presence of two Y^{3+} ions stabilizes the tetragonal crystal structure, creating an oxygen vacancy and a matrix deformation. Ionic species originating from water, such as OH^- and/or potentially H^+ and O^{2-}, diffuse into the material and fill the oxygen vacancies, thereby destabilizing the tetragonal structure and initiating a transformation to the monoclinic structure. This mechanism explains the onset of the transformation on the material surface.

The process initiates through the formation of a martensitic variant within a tetragonal zirconia grain, which is known as the uplift area (Kelly and Rose 2002). After nucleation, the transformation propagates throughout the grain until the grain boundary is reached, which acts as a barrier to further transformation. This phenomenon induces stresses in neighboring grains, initiating their surface transformation (Deville et al. 2004; Munoz-Tabares et al. 2011). The progression through the material is essentially based on the propagation of water through the microcracks in the material.

The following factors are reported to influence LTD (Kelly and Denry 2008).

- The grain size, which is a function of the sintering temperature (2 h sintering at 1350 °C yields a 0.3 µm grain size, whereas a sintering temperature of 1650 °C yields a grain size in excess of 2 µm) (Gremillard et al. 2004; Hallmann et al. 2011; Li and Watanabe 2005; Sato and Shimada 1985). Large grains lead to a greater aging kinetics. This may be due to indirect effects: larger grains are cubic grains, which can induce an yttrium depletion in neighboring tetragonal grains (see below). In addition, the grain boundaries constitute obstacles to transformation (but do not prevent from its propagation), and small grains have more interface energy with others, which can increase stability.
- The stabilizing oxide (Y_2O_3) concentration. Grains containing a reduced amount of yttrium oxide exhibit a reduction in stability and are, therefore,

more susceptible to aging by water. Sato and Shimada showed that increasing the amount of yttrium oxide increases the stability of the tetragonal phase (Sato and Shimada 1985). However, the toughness of the material decreases as the yttria concentration increases (Chevalier et al. 2009). A Y_2O_3 concentration of 3 mol% is considered the correct amount for optimizing the mechanical performance.

- The stabilizing oxide (Y_2O_3) distribution. An inhomogeneous distribution of Y^{3+} ions can yield randomly dispersed cubic grains exhibiting high Y^{3+} concentrations. However, their development occurs at the expense of neighboring grains, which consequently contain less yttria and exhibit increased metastability and are, therefore, more susceptible to transformation (Chevalier et al. 2004).
- Yttrium and alumina co-doping. The presence of small amounts of alumina results in slower aging kinetics (Ross et al. 2001; Tsubakino et al. 1991). Indeed, alumina promotes solid phase sintering and then good material density at lower sintering temperature and smaller grain size. In addition, alumina segregating at the grain boundaries modifies the diffusion of OH^- ionic species and therefore by itself modifies the aging kinetics.
- The crystallography. The cubic phase is immune to aging, while the tetragonal phase is sensitive to LTD, all the more it has less yttria. The cubic phase concentration increases with the yttria content and the sintering temperature (Chevalier et al. 2004).
- The presence of internal stresses. These stresses may be introduced through surface treatments, such as sandblasting or milling, and act as nucleation sites for LTD (Deville et al. 2006; Guazzato et al. 2005).

Due to the high variability of the materials tested, the protocols used, and the risk of bias in various studies (Pereira et al. 2015), results regarding the influence of LTD on material strength are controversial. Some studies have shown that LTD significantly decreases (Ban et al. 2008; Cattani-Lorente et al. 2011; Kim et al. 2009; Kohorst et al. 2012) or increases (Alghazzawi and Janowski 2015; Siarampi et al. 2014) the flexural strength of several Y-TZP dental ceramics, whereas others have reported that it does not exert any

influence (Alghazzawi et al. 2012; Amaral et al. 2013; Bergamo et al. 2016; Borchers et al. 2010; Camposilvan et al. 2018; Cotes et al. 2014; Pereira et al. 2016). However, a systematic review published in 2015 concluded that LTD induced through autoclave aging (at an aging duration > 20 h, a pressure ≥ 2 bars, and a temperature of 134 °C) significantly increases the *m*-phase content and decreases the flexural strength of the first- and second-generation 3Y-TZP dental ceramics (Pereira et al. 2015). Certain studies reported that LTD was responsible for a significant decrease in flexural strength in samples where 50% of the surface was transformed (Ban et al. 2008; Kim et al. 2009; Pereira et al. 2015). Recent international standardization guidelines for zirconia (Iso 13356-2015. Implants for surgery – ceramic materials based on yttria-stabilized tetragonal zirconia (y-tzp) 2015) (ISO 13356- 2015) have established that the monoclinic phase content should not exceed 25% in Y-TZP implants, after aging in an autoclave at 134 °C and 2 bars for 5 h, to be considered suitable for biomedical purposes.

3. NOVEL GENERATIONS OF DENTAL ZIRCONIA

Currently, three generations of dental zirconia materials can be identified (Zhang and Lawn 2018) (Table 1.1). The original zirconia material (3Y-TZP), which is used to manufacture frameworks for veneered restorations, constitutes the first generation. The distinguishing characteristics of this generation are the low cubic phase content (typically <15 vol.%) and the resultant high strength (1200 MPa) and toughness. However, esthetic drawbacks, owing to its high opacity and birefringence, limit its use. Translucency was improved in the second generation through a decrease in the alumina content and/or an increase in the sintering temperature to reduce porosity while increasing the grain size (Denry and Kelly 2014). Impurities exhibiting a different refractive index than that of zirconia (for example, alumina) can reduce the translucency of Y-TZP (Zhang et al. 2012). On the other hand, an increase

Zirconia	Composition	Properties	Applications
First-generation	3Y-TZP (<15% cubic phase)	High opacity/ refractive index High strength & toughness 1000–1500 MPa flexural strength (manufacturers' data)	Framework for veneered restorations
Second- generation: Translucent	3Y-TZP (<15% cubic phase) ↘ Alumina Sintering at higher temperature to reduce porosities ↗ Metastability	↗ Translucency ↗ LTD 900–1300 MPa flexural strength (manufacturers' data)	Monolithic (or veneered) posterior restorations
Third-generation: Highly translucent	4Y-PSZ (>25% cubic phase) 5Y-PSZ (>50% cubic phase) ↗ Grain size: 1.5 µm ↘ Metastability	↗ Translucency ↘ LTD ↘ Strength & toughness 400–1000 MPa flexural strength (manufacturers' data)	Monolithic (or veneered) single-unit restorations Max three elements- bridges

Table 1.1 *Characteristics of the current generations of dental zirconia materials in terms of composition, properties, and applications.*

in the grain size results in a reduction in the number of grain boundaries, which provide a natural barrier to the transmission of light. Optical property improvements facilitate the fabrication of posterior (molars and premolars)

monolithic restorations; however, modifications to material composition and microstructure may reduce the resistance to LTD owing to increased metastability (Denry and Kelly 2014; Lawson et al. 2014; Preis et al. 2015). Initially, the use of the tetragonal zirconia phase was predominant in the field of dental prostheses. More recently, novel (third-generation) high-translucent varieties have been developed that utilize a higher proportion of yttria, thus in turns of both an increase in the cubic phase content and a decrease in the tetragonality of the *t* phase. The tetragonal phase is anisotropic; therefore, its optical behavior depends on the alignment of the crystalline axes. This anisotropy can induce a double refraction phenomenon known as birefringence (where the penetrating light ray is divided into two). The double refraction associated with the high refractive index of $Zr0_2$ induces significant light scattering, significantly limiting the component's translucency. Conversely, the cubic phase is isotropic, and its optical behavior is homogeneous across all crystalline axes. Therefore, there is no birefringence phenomenon observed in these phases, resulting in significantly lower light losses through dispersion and, consequently, greater translucency. It is possible to obtain zirconia containing a higher cubic phase proportion at room temperature by increasing the yttrium oxide content, thus generating a higher translucency over conventional varieties. Therefore, esthetic anterior (incisors and canines) monolithic restorations are now feasible through the utilization of third-generation zirconia. These restorations exhibit increased translucency and LTD resistance as a result of the higher cubic phase content (yttria-partially stabilized zirconia (Y-PSZ) with 4 or 5 mol%: 4Y-PSZ contains more than 25% of the cubic phase and 5Y-PSZ more than 50%). However, as the cubic phase does not undergo a stress-induced transformation, its mechanical properties are significantly reduced in comparison to the two preceding generations (<700 MPa flexural strength for 5Y-PSZ). Therefore, their applications are limited, particularly with respect to bridge fabrication, and the loss of strength makes them less able to compete with glass-ceramics, which also exhibit increased adhesion to tooth tissues. The trade-off between translucency and mechanical resistance is significant (Camposilvan et al. 2018; Zhang and Lawn 2018).

4. CLINICAL BACKGROUND WITH MONOLITHIC ZIRCONIA-BASED RESTORATIONS

The majority of publications reporting the clinical uses of zirconia monolithic prostheses describe restorations fabricated from second-generation zirconia (Batson et al. 2014; Bomicke et al. 2017; Cardelli et al. 2016; De Angelis et al. 2019; Diéguez-Pereira et al. 2020; Habibi et al. 2020; Hansen et al. 2018; Hartkamp et al. 2017; Kitaoka et al. 2018; Konstantinidis et al. 2018; Lohbauer and Reich 2017; Mangano et al. 2019; Miura et al. 2020; Moscovitch 2015; Papaspyridakos et al. 2020; Pathan et al. 2019; Pol et al. 2020; Pontevedra et al. 2020; Rammelsberg et al. 2020; Rojas Vizcaya 2016; Sakornwimon and Leevailoj 2017; Solá-Ruiz et al. 2020; Stober et al. 2016; Sulaiman et al. 2016; Tang et al. 2019; Worni et al. 2017).

With respect to implant-supported FPDs, Cardelli et al., Papaspyridakos et al., and Pol et al. presented encouraging short-term results. The first two studies presented results obtained from two patients rehabilitated with monolithic zirconia full-arch prostheses fixed on implants after 1 year (Cardelli et al. 2016) and 2 years (Papaspyridakos et al. 2020). The third was a prospective case study involving 56 three-unit FPDs (Pol et al. 2020). Vizcaya reported excellent results of a 2–7 year retrospective study pertaining to 20 double full-arch prostheses (Rojas Vizcaya 2016), as did Diéguez-Pereira et al. in a retrospective study of up to 5 years by using 14 full-arch and 35 FPDs (Diéguez-Pereira et al. 2020). These encouraging results can be attributed to the fact that splinting implants improve the stress distribution (Vigolo et al. 2015). In a cohort study by Rammelsberg et al. (Rammelsberg et al. 2020), the survival rate of 68 FPDs was 100%; however, two chipping events were observed after a mean observation period of 2.1 years, which is unusual in a high-strength material.

For implant-supported crowns, a 100% survival rate over 3 years was reported for 19 screw-retained restorations (De Angelis et al. 2019), and a 100% survival

rate was reported in a retrospective study of 33 crowns up to 5 years after implantation (Diéguez-Pereira et al. 2020).

Tooth-supported crowns have been shown to exhibit high survival rates and low complication rates (Mazza et al. 2021), with the majority of complications being attributed to endodontic problems (Bomicke et al. 2017), debonding (Bomicke et al. 2017; Solá-Ruiz et al. 2020), fractures (Hansen et al. 2018; Miura et al. 2020), and chipping (Hansen et al. 2018).

For tooth-supported FPDs, Pontevedra et al. reported a 100% survival rate after 28 months (Pontevedra et al. 2020), whereas Habibi et al. reported a 96.7% survival rate after 3 years, recording two endodontic problems, one debonding and one fracture (Habibi et al. 2020).

It must be highlighted that high-risk patients, particularly those affected by bruxism, were not included in clinical studies, except in the study conducted by Hansen et al. (Hansen et al. 2018). However, bruxism is reported to be highly prevalent (Kuhn and Türp 2018) with manufacturers often recommending high-strength monolithic zirconia restorations even for this condition. The inclusion of patients with bruxism in clinical studies is rare but crucial in assessing the viability of new materials and techniques in prosthodontics.

5. MONOLITHIC ZIRCONIA RESTORATIONS AND THEIR WEAR ISSUES

Material and antagonistic tooth wear were of primary concern for clinicians when monolithic zirconia restorations were first introduced. The considerable hardness of zirconia was suspected to induce a high wear rate in opposing teeth. However, several *in vitro* studies have shown that antagonist enamel wear against zirconia is less than with felspathic ceramics, glass-ceramics, and

natural teeth (D'Arcangelo et al. 2018; Jung et al. 2010; Nakashima et al. 2016; Sripetchdanond and Leevailoj 2014; Zhang et al. 2019). The fine and homogeneous microstructure of zirconia, which is composed of grains of approximately 0.3 µm, limits the material's abrasiveness. In fact, wear is a complex phenomenon in which the hardness is not the only parameter. On the other hand, some authors reported that LTD was suspected to increase the surface roughness of Y-TZP ceramics (Hernigou and Bahrami 2003; Santos et al. 2004). Nonetheless, in a recent systematic review and meta-analysis, LTD simulated by steam autoclave aging was not shown to affect its surface roughness (Yang et al. 2020). In a recent meta-analysis of four studies (Solá-Ruíz et al. 2020), a maximum vertical wear ranging between 51.9 and 204.0 µm for the antagonistic teeth was reported after a follow-up time of 6–24 months. The maximum vertical wear in each tooth was calculated from the mean of 10 depth values taken from the area surrounding the maximum recorded wear in each differential scan area. A combination of studies based on a random-effects model indicated a mean maximum wear of 95.5 µm, with a confidence interval of 95% (79.6–111.3), which is greater than the wear generated by natural teeth (Sagirkaya et al. 2012; Stober et al. 2016).

Several factors influence antagonistic tooth wear: the surface treatment of the monolithic zirconia restoration (glazed or polished), the position of the restoration (wear is more significant in molars than in premolars), and gender (wear is less frequent in women than in men). In a recent systematic review of *in vitro* and *in vivo* studies, investigations pertaining to the influence of the finishing procedure showed that polishing should be preferred over glazing or veneering (Gao et al. 2021). Several authors (Esquivel-Upshaw et al. 2018; Lohbauer and Reich 2017; Mundhe et al. 2015) measured enamel wear using polished zirconia, whereas others (Stober et al. 2016) evaluated enamel wear using glazed zirconia; however, the wear rates of polished and glazed zirconia were not compared in any study. The glazed surface exhibits an increased roughness in comparison to zirconia (Rupawala et al. 2017), and

when it is worn away, the underlying raw zirconia is often rougher than polished zirconia (Janyavula et al. 2013; Sripetchdanond and Leevailoj 2014). With respect to the zirconia material wear, *in vitro* studies comparing the wear behavior of different restorative materials (zirconia, glass-ceramics, glass-infiltrated zirconia/alumina, feldspathic, polymer-infiltrated ceramic network materials, and dental composites) revealed that zirconia shows the lowest material wear (Albashaireh et al. 2010; Borrero-Lopez et al. 2019; Dupriez et al. 2015; Kruzic et al. 2018; Miyazaki et al. 2013; Mörmann et al. 2013; Preis et al. 2011; Rosentritt et al. 2012; Santos et al. 2018; Zhang et al. 2019). The clinical studies included in the previously mentioned meta-analysis estimated wear between 38.4 µm and 145.0 µm after a follow-up period between 6 and 24 months. A combination of the results obtained from the random-effect-model studies estimates a maximum wear of 58.4 µm, with a corresponding confidence interval of 95% (45.4–71.5). However, *in vivo* material or tooth tissue wear measurements remain a challenge, particularly with respect to the resolution required to measure vertical variations of <10 µm. The "replica technique" is frequently used to model wear. This technique utilizes a polyvinylsiloxane impression to fabricate a plaster or resin tooth, which is then used to perform wear measurements (Esquivel-Upshaw et al. 2018; Lohbauer and Reich 2017; Mundhe et al. 2015; Pathan et al. 2019; Scherrer et al. 2007; Stober et al. 2016). However, the use of a replica does not provide an ideal solution as it induces a loss of accuracy. In addition, many techniques, each exhibiting varying degrees of performance, can be used to measure material wear, such as surface scanning and the use of matching software, with the choice of protocol influencing the results and measurement chain capacity.

6. MONOLITHIC ZIRCONIA RESTORATIONS AND THE LTD ISSUE

In the early 2000s, LTD of 3Y-TZP ceramics induced significant clinical consequences in orthopedic devices such as, the fracture of approximately 600 to 800 zirconia heads within two years of implantation (Chevalier et al. 2009). Seven batches of the Prozyr® material, manufactured using tunnel furnaces and suspected to exhibit increased LTD susceptibility, were involved (Chevalier 2006). Numerous lab scale studies pertaining to the effects of LTD on implants have been conducted (Cattani-Lorente et al. 2011; Chevalier et al. 2007; Douillard et al. 2012; Gremillard et al. 2018; Gremillard et al. 2013; Munoz-Tabares et al. 2011; Samodurova et al. 2015). Several assessments with respect to *in vivo* aging were performed through retrospective explant analyses. The results of these confirmed many of the features discussed at the lab scale, and also recorded surface degradation on retrieved zirconia hip implants (Catledge et al. 2003; Chevalier 2006; Fernandez-Fairen et al. 2007; Haraguchi et al. 2001; Santos et al. 2004; Tateiwa et al. 2020) and/or related strong osteolysis (Allain et al. 1999; Hernigou and Bahrami 2003; Norton et al. 2002). The results showed that LTD was responsible for a portion of the clinical failures resulting from the use of zirconia in orthopedics. The Prozyr® heads broke as a result of crack propagation which initiated in the micro-cracked, transformed region, which was loaded in tension. Many retrieved heads exhibited extensive grain pull-out, which resulted in material debris around the implant, chronic inflammation, and in the worst cases, osteolysis. These issues, in particular the "Prozyr® failures", had a catastrophic impact on the use of Y-TZP in the field of orthopedics, despite contrasting results from zirconia heads of different compositions, which exhibited strong performances and low wear rates (Caton et al. 2004; Wroblewski et al. 2004). Therefore, research in the orthopedic field has concentrated on the development of alternative materials, particularly alumina-zirconia composites, to improve the resistance to LTD (Chevalier et al. 2007; Deville et al. 2005). In contrast with its abandonment in orthopedics, the use of Y-TZP has signi-

ficantly increased in the dental prosthesis field owing to its increased optical and biocompatibility performance when compared with metal alloys, alongside its high mechanical resistance when compared with glass-ceramics. Although 3Y-TZP has been widely used in dental applications (Makhija et al. 2016), LTD has rarely been investigated *in vivo* during clinical studies. The primary investigative route has been *in vitro* simulation using steam autoclave aging (Arata et al. 2014; Inokoshi et al. 2015; Lughi and Sergo 2010). In fact, the principles and mechanisms behind LTD are quite well-described; however, there are still debates as to the suitability of Y-TZP in dental applications. There is particular concern with respect to the so called "translucent" zirconia materials (second generation) which were developed to improve the esthetic appearance of prostheses and are supposed to exhibit increased metastability (Zhang and Lawn 2018) (see section 3).

Indeed, *in vitro* autoclave aging conditions differ significantly from those experienced in the body (Pezzotti et al. 2017). The exposure to saliva (an ionic solution), acidic beverages, and constant changes in pH are not taken into account (Alghazzawi et al. 2012; Swain 2014), and the mechanical stress due to masticatory forces (notably tribological effects) is not considered, despite these effects being shown to accelerate Y-TZP degradation, as recorded in both *in vitro* (Wei and Gremillard 2018) and *in vivo* (explant) analysis of zirconia hip prostheses (Douillard et al. 2012; Tateiwa et al. 2020). Many researchers have analyzed LTD after sample immersion in water at 37 °C for 1–4 years (Cattani-Lorente et al. 2016; Keuper et al. 2014; Pereira et al. 2016), whereas others have performed mechanical cycling on dental crowns in water at 37 °C to simulate a year's wear (Bergamo et al. 2016). However, the simulation of oral function using a mechanical simulator depends on a significant number of parameters such as the direction and strength of applied constraints, speed and number of chewing cycles, elimination of worn particles, sample form (flat surface or natural cusps), and antagonist nature (stainless steel or steatite balls). To date, there is no effective standardization, and, therefore, results issued from different laboratories cannot be

compared (Heintze 2006). Recently, Schepke et al. reported the results of the examination and analysis of 44 implant abutments fabricated from first-generation zirconia material 1 year after their implantation. Raman analysis of the zirconia connection, which was screwed into the titanium endo-osseous implant, did not reveal any *t-m* transformation. However, the abutment connection is not exposed to masticatory stresses or to the surrounding oral environment (Schepke et al. 2019). In 2017, Miragaya et al. studied the influence of intraoral aging on the *t-m* phase transformation of first- and second-generation Y-TZP dental ceramics (Lava Frame and Lava Plus, respectively) and determined the impact of this response on their mechanical properties. After the baseline analysis, the specimens were attached to personalized intraoral resin appliances, exposed to the oral cavities of 20 subjects for 60 days, and then analyzed again. Intra-oral aging for 60 days induced the *t-m* phase transformation and significantly decreased the mechanical properties of both generations of Y-TZP dental ceramics. However, it should be noted that the samples were not subjected to mechanical stresses.

In conclusion, future work focused on the monitoring of the LTD process in the body environment would be of particular interest and significance, with the oral cavity providing an easy to access, interesting, and experimental environment. Long-term follow-up of zirconia monolithic restorations would facilitate the investigation of the intraoral development and kinetics of LTD, and the influence of masticatory mechanical stress on this process could be evaluated.

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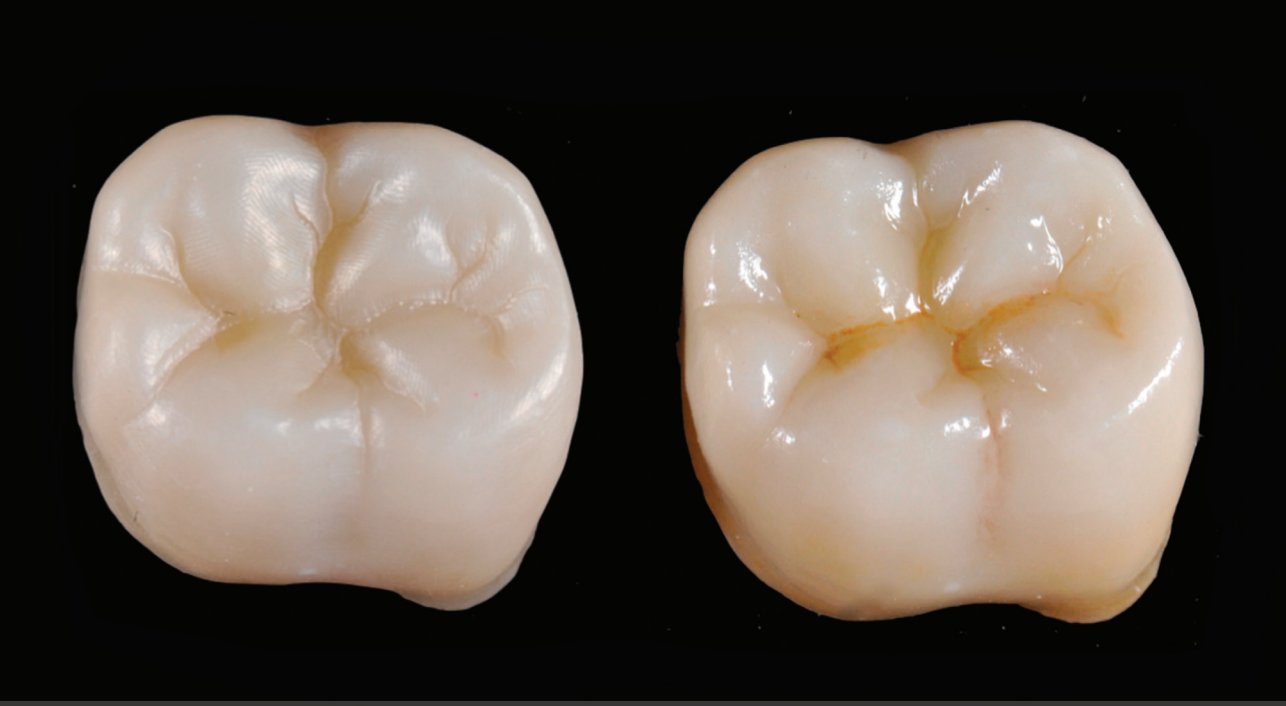


2

Objectives

1. OBJECTIVES

The main objective of this thesis was to contribute to the understanding of intraoral development and kinetics of LTD in second-generation 3Y-TZP monolithic prostheses and the influence of occlusal stress and glaze protection on this process.

Secondary objectives include the investigation of restorations' general clinical behavior and material wear.

The strategy was based on the following assumptions developed in the introduction:

- Clinical studies on zirconia-based restorations (ZBR) have indicated that chipping of the veneering ceramic is an important cause of failure. If the manufacturing process was thoroughly investigated to explain this problem, the influence of clinical risk factors, particularly intrinsic factors related to the patient, were not studied because, for example, most clinical studies excluded bruxers.
- In addition, the introduction of computer-aided design and manufacturing monolithic zirconia dental prostheses to improve prosthesis mechanical resistance raises the issue of material low-temperature degradation (LTD), a well-known problem with zirconia hip prostheses, which could be promoted in second-generation materials. LTD has been sparsely investigated *in vivo*, and the influence of mechanical loading such as masticatory stresses or the effect of the glaze layer on dental prostheses has never been studied.
- Publications reporting *ex vivo* quantitative measurements of dental zirconia wear showed negligible wear of zirconia surfaces and less abrasive effects on antagonist teeth than other ceramic materials. However, the literature reports different methods to clinically evaluate dental tissue and material wear, and most of the measurements are performed on replicas, which can generate significant imprecision in wear evaluation and counteract thorough surface observations.

- The literature is sparse regarding the clinical outcomes of zirconia monolithic prostheses, and patients presenting bruxism are generally excluded from studies.

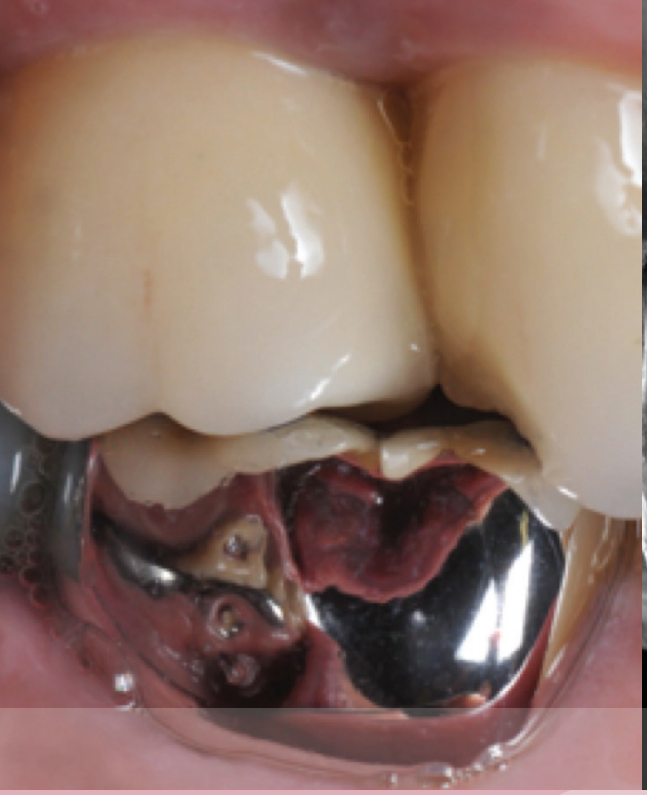
Therefore, the specific aims of this thesis are as follows:

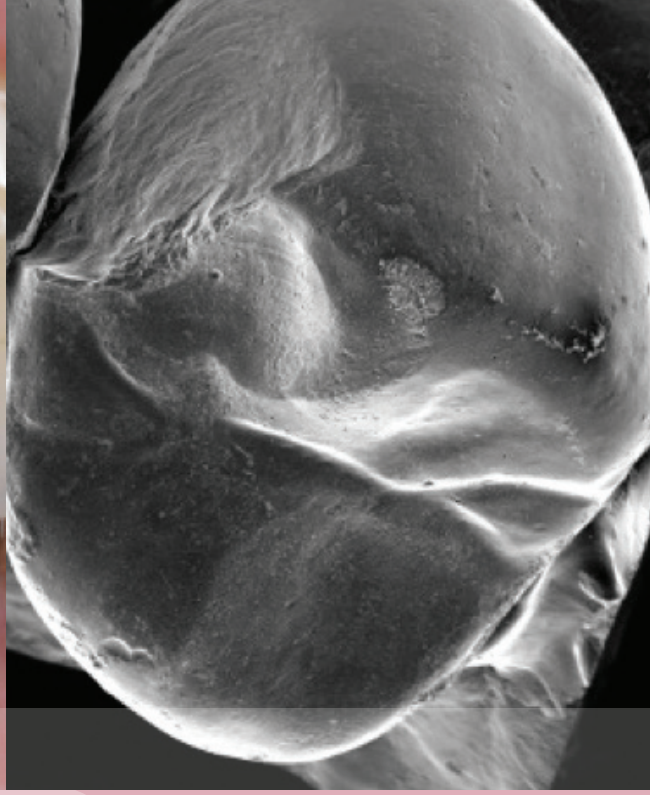
1. To retrospectively evaluate ZBR performed in the Department of Fixed Prosthodontics of the University of Liege and to correlate fractures with clinical parameters that can influence external stresses.
2. To investigate the intraoral development and kinetics of LTD in the second generation, 3 mol.% yttria-doped tetragonal zirconia polycrystal monolithic prostheses, as well as the influence of masticatory mechanical stress and glaze layer on it.
3. To investigate the clinical outcomes of second-generation zirconia restorations, including patients with bruxism clinical signs and the material wear process.

In order to achieve the second and the third objectives, two preliminary work packages were developed. They aimed, respectively:

- To develop an original prospective clinical study protocol that includes *ex vivo* analyses of the restoration to investigate (1) intraoral LTD process of second-generation zirconia restorations on teeth and implants with Raman spectroscopy, (2) biological, functional, and esthetic clinical outcomes in patients with and without clinical signs of bruxism, and (3) material wear with 3D laser profilometry and glaze wear using scanning electron microscopy imagery.

- To systematically review the different methods used for wear measurement of dental tissues and materials in clinical studies, their relevance and reliability in terms of accuracy and precision, and the performance of the different steps of the workflow taken independently.





3

Clinical risk factors related to failures with zirconia-based restorations: An up to 9-year retrospective study

Vinciane Koenig [1]
Alain J. Vanheusden [1]
Stéphane O. Le Goff [2]
Amélie K. Mainjot [1, 2]

[1] *Department of Fixed Prosthodontics, Institute of Dentistry, University of Liège Hospital (ULg, CHU, LIMARC), 45 Quai G. Kurth, Liège, 4020, Belgium.*

[2] *Unité de Recherches Biomatériaux Innovants et Interfaces (URB2I-EA4462), Université Paris Descartes, Sorbonne Paris Cité, Faculté de Chirurgie Dentaire, Paris, France.*

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ABSTRACT

Objectives: The first objective of this study was to retrospectively evaluate zirconia-based restorations (ZBR). The second was to correlate failures with clinical parameters and to identify and to analyse chipping failures using fractographic analysis.

Methods: 147 ZBR (tooth- and implant-supported crowns and fixed partial dentures (FPDs)) were evaluated after a mean observation period of 41.5 ± 31.8 months. Accessorily, zirconia implant abutments (n = 46) were also observed. The technical (USPHS criteria) and the biological outcomes of the ZBR were evaluated. Occlusal risk factors were examined: occlusal relationships, parafunctional habits, and the presence of occlusal nightguard. SEM fractographic analysis was performed using the intra-oral replica technique.

Results: The survival rate of crowns and FPDs was 93.2%, the success rate was 81.63% and the 9-year Kaplan–Meier estimated success rate was 52.66%. The chipping rate was 15% and the framework fracture rate was 2.7%. Most fractographic analyses revealed that veneer fractures originated from occlusal surface roughness. Several parameters were shown to significantly influence veneer fracture: the absence of occlusal nightguard (p = 0.0048), the presence of a ceramic restoration as an antagonist (p = 0.013), the presence of parafunctional activity (p = 0.018), and the presence of implants as support (p = 0.026). The implant abutments success rate was 100%.

Conclusions: The results of the present study confirm that chipping is the first cause of ZBR failure. They also underline the importance of clinical parameters in regards to the explanation of this complex problem. This issue should be considered in future prospective clinical studies.

Clinical significance: Practitioners can reduce chipping failures by taking into account several risk parameters, such as the presence of a ceramic restoration as an antagonist, the presence of parafunctional activity and the presence of implants as support. The use of an occlusal nightguard can also decrease failure rate.

Keywords: zirconia, chipping, fractography, fixed dental prostheses, risk factors, technical complications.

1. INTRODUCTION

In prosthodontics, porcelain-fused-to-metal (PFM) restorations have passed the test of time and are recognized as predictable and reliable clinical solutions for fixed prostheses. Nevertheless, since the eighties, the increasing aesthetic demand in dentistry has driven the development of various ceramic core materials. Introduced a decade ago as an alternative to metal, yttria-tetragonal zirconia polycrystal (Y-TZP) exhibits transformation-toughening properties, which give it high strength and tenacity in comparison with other ceramic core material [1]. Unfortunately, despite Y-TZP frameworks mechanical properties, clinical reports about zirconia-based crowns and fixed partial dentures (FPDs) have indicated a high rate of short-term failures, which are related to cohesive fracture of the veneering ceramic (chipping). In the systematic review of Schley et al. [2], the mean estimated 5-year survival rate for FPDs was 94.3%, while the 5-year complication free rate was only 76.4%. Chipping is the most frequent technical complication and is reported to be significantly higher than for PFM restorations [3–5]. The short-term crowns and FPD chipping rate varies from 0 to 88.9%, the weighted (by sample size) average of 32 studies being 12.3% (Table 3.1) [4–35], while a 2.9% chipping rate is reported for PFM restorations [2,36–38]. Some studies distinguish minor chipping, which can be simply polished and does not require removal from the restoration, from major chipping and delaminations. However delaminations, which imply a fracture at the interface between veneer and zirconia, are not easy to diagnose intra-orally, since high magnification observation is required to confirm the interfacial rupture and the framework exposition. In 2007, Anusavice et al. pointed out the necessity to standardize the diagnosis of veneer fracture, but today this is still lacking even with PFM restorations, as well as the analysis of this type of clinical failure [39]. The authors particularly recommended the development of a comprehensive classification system for identifying failures and the utilization of fractographic analysis to identify the fracture origin. The same year, Scherrer et al. [40] demonstrated the benefit of the replica technic for the fractographic analysis of failed ceramic restorations. This technic allows

Study	Follow-up period (years)	Sample size	Veneering porcelain fracture (%)
Larsson et al. (2013) [6]	8	9	88.9
Rinke et al. (2013) [7]	7	80	28.8
Zembic et al. (2012) [8]	5.6	18	0
Ortorp et al. (2012) [9]	5	143	3
Vigolo et al. (2012) [5]	5	39	7.7
Lops et al. (2013) [10]	5	37	10.8
Burke et al. (2013) [11]	5	33	24.2
Sailer et al. (2007) [12]	5	33	15
Schmitter et al. (2012) [13]	5	30	26.7
Schmitt et al. (2012) [14]	5	25	28
Molin et Karlsson (2008) [15]	5	19	0
Sagirkaya et al. (2012) [16]	4	107	0.9
Peláez et al. (2012) [4]	4	20	10
Salido et al. (2012) [17]	4	17	29.1
Beuer et al. (2010) [18]	3	68	7.4
Tinschert et al. (2008) [19]	3	65	6
Rinke et al. (2013) [20]	3	52	5.8
Sailer et al. (2009) [21]	3	36	33.4
Edelhoff et al. (2008) [22]	3	21	9.5
Beuer et al. (2009) [23]	3	21	0
Schmitt et al. (2010) [24]	3	17	5.9
Papaspyridakos et al. (2012) [25]	3	16	31.3
Raigrodski et al. (2006) [26]	2.5	20	25
Pospiech et al. (2003) [27]	2	38	2.6
Esquivel-Upshaw et al. (2013) [28]	2	36	16.7
Vult Von Steyern et al. (2005) [29]	2	20	15
Tsumita et al. (2010) [30]	2	21	14.3
Cehreli et al. (2009) [31]	2	15	0
Ohlmann et al. (2012) [32]	2	10	20
Bornemann et al. (2003) [33]	1.5	59	3.3
Ohlmann et al. (2008) [34]	1	30	13
Larsson et al. (2006) [35]	1	13	54
Weighted (by sample size) average			12.3

Table 3.1 *List of clinical trials conducted in Y-TZP-based restorations.*

the identification of the crack's origin and its direction of propagation, analysing fractographic markers such as hackle lines, arrest lines or wake hackles on an epoxy replica of the restoration.

The chipping mechanism is complex and not well understood in such complex geometries as dental crowns and bridges. Numerous *in vitro* studies about fracture strength, crack propagation, and veneer-zirconia adhesion have been published. But there are both advantages and limitations to flexure, tensile and shear tests. These tests are particularly not easy to interpret and a lack of standardized procedures has been described [39]. Moreover, the source of cracks in dental ceramics is multifactorial and hard to define: thermal stress caused by thermal incompatibility during the manufacturing process [41], or an inadequate framework design [42] are examples which have been reported to promote veneer fracture.

However, it is known that in all cases cracks form and propagate when the tensile stress within the ceramic exceeds the tensile strength [39]. The tensile stress at a specific location of the ceramic is the sum of external and residual stresses. Residual stresses are "locked-in" stresses present within the veneer and the framework from the time when the veneering ceramic solidifies. These stresses exist and persist within the material without the application of any external load but will add to its effect [43]. The residual stresses can cause immediate or delayed cracking of the ceramic. The cooling rate, the coefficient of thermal expansion (CTE) of the veneering ceramic, as well as the core-veneer thickness ratio have been reported to influence these stresses [44-46].

On the other hand, external stress is generated within the structure by applied loads, i.e. mostly during function and mastication. The amount of external stress is influenced by clinical parameters such as overloading due to bruxism. Nevertheless, most clinical studies include bruxism in their exclusion criteria, and therefore a potential limitation of this all-ceramic system has not been investigated clinically. In the study by Papaspyridakos [25], the parafunctional

habits, the nature of the antagonist and the absence of nocturnal bruxism splint were associated with chipping.

The first objective of this study was to retrospectively evaluate zirconia-based restorations (ZBR) performed in the Department of Fixed Prosthodontics of the University of Liege. The second objective was to identify and analyse veneer fracture failures, using fractographic analysis with the intra-oral replica technique and correlating fractures with clinical parameters that can influence external stress.

2. MATERIALS AND METHODS

2.1 Study design

This is a retrospective record evaluation and a clinical examination of patients treated with ZBR. The patients were recruited in the Department of Fixed Prosthodontics of the University of Liege and were treated by four experienced practitioners. ZBR analysed in this study included implant abutments, conventionally cemented crowns and FPDs on natural teeth and implants, and screw-retained crowns and FPDs on implants.

Among the 90 patients recalled, 65 agreed to participate in the study. The patients were informed of the purposes of the study and consent was obtained before examination.

The ZBR (*n* = 193) were performed during the follow-up period from May 2003 to January 2012. Three different dental laboratories were involved in the manufacturing process of respectively 8, 40 and 99 crowns and FPDs.

2.2 Patient record registrations

Specific data were collected regarding the following parameters: sex, age, restoration design (type, number of elements), restoration brand, dental laboratory, abutment and antagonist teeth characteristics, and type of cement used. For implant abutments the different types of connections (internal or external) were recorded.

2.3 Occlusal risk factors evaluation

Occlusal relationships were characterized as favourable or unfavourable based on the clinical examination. Class III or class II.2 malocclusion, anterior or posterior crossbite, edge to edge or open bite, were considered as unfavourable occlusal relationships.

The presence of parafunctional habits was recorded if the patient related grinding or clenching habits, masticatory muscle discomfort, or if abnormal wear facets were observed on teeth. The use of an occlusal nightguard was noted. Finally, when a fracture of the veneering porcelain was detected, the occlusal contact points and facets were examined in order to determine whether the fracture was located on an occlusal contact or guidance.

2.4 Restorations evaluation

2.4.1 Biological

The biological evaluation compared periodontal measurements on the abutment tooth (test) and an analogous, contralateral, tooth without restoration (control). This evaluation included probing pocket depth (PPD), probing attachment level (PAL), absence or presence of plaque using the

plaque control record (PCR), and bleeding on probing (BOP). The presence of secondary caries and endodontic complications was assessed and radiographs were performed.

2.4.2 Technical

For the evaluation of the technical performance of ZBR, the United States Public Health Service (USPHS) criteria were used (Table 3.2). Variables Alpha and Bravo were defined as successes, whereas variables Charlie and Delta were defined as failures. An outcome was rated Alfa (A) when no problem occurred, Bravo (B) when small but clinically acceptable defects were found,

	Alpha (A)	Bravo (B)	Charly (C)	Delta (D)
Framework fracture	No fracture of framework			Fracture of framework
Veneering fracture	No fracture	Fracture, but polishing possible	Chipping down the framework	New reconstruction is needed
Occlusal wear	No occlusal wear on reconstruction or on opposite teeth	Occlusal wear on reconstruction or on opposite teeth < 2mm	Occlusal wear on reconstruction or on opposite teeth > 2mm	New reconstruction is needed
Marginal adaptation	No probe catch	Slight probe catch, but no gap	Gap with some dentine or cement exposure	New reconstruction is needed
Anatomical form	Ideal anatomical shape, good proximal contact	Slightly over- or under-contoured weak proximal contact	Highly over- or under-contoured open proximal contact	New reconstruction is needed

Table 3.2 *USPHS criteria.*

Charlie (C) when the defects reached a level that was no longer clinically acceptable, and Delta (D) when the ZBR had to be replaced [21]. The technical performance of FPDs was evaluated on the basis of the worst result among the different abutments. Loss of retention of luted restorations and unscrewing of implant abutments or screw-retained restorations were noted. Finally clinical pictures of the restorations were performed.

2.5 Replica preparation and fractographic analysis

If an untreated fracture of the veneering ceramic (B, C or D) was detected, a double-mix impression of the ZBR surface was performed with a high- and a low-viscous A-silicone impression material. The fracture surface was first cleaned with a cotton pellet and alcohol and was then rinsed and thoroughly air-dried [40]. The replicas were produced using a quadrafunctional hydrophilic siloxane impression material (Aquasil ULV, Dentsply De Trey, Konstanz, Germany), the low viscosity material being syringed onto the fractured surface and over the occlusal surface of the crown. The impressions were cast with cold mounting epoxy resin (Epofix Resin, Struers, Ballerup, Denmark). After setting, the model was sectioned to isolate the restoration replica to be analysed, which was gold-coated for scanning electron microscopy (SEM). Moreover hard plaster models (GC Fujirock EP Super Hard Plaster, GC Europe, Leuven, Belgium) of each maxilla were performed to observe the occlusal relationships between the two arches.

The fractographic analysis was performed using a JSM-6400 Scanning Electron Microscope (JEOL Limited, Tokyo, Japan). The interpretation of fracture patterns was based on the descriptions by Scherrer et al. [40], particularly to determine the origin and direction of the crack propagation. The fracture extent was also explored, distinguishing cohesive fracture from delamination, i.e. adhesive fracture between veneer and zirconia framework.

2.6 Patient satisfaction level

The patients were asked to score their ZBR in terms of aesthetic and functional outcomes on a 10-point visual analogue scale (VAS) with the endpoints extremely dissatisfied (0) and extremely satisfied (10).

2.7 Statistical analysis

Descriptive statistics were applied to the data. Analysis of the survival rate of the ZBR was performed by means of Kaplan-Meier survival statistics followed by a log-rank test. The USPHS evaluation was compared using the chi-square test. For the comparison of PPD, PAL, PCR and BOP between test and control teeth, Wilcoxon, Kappa and t-student tests were used. The restoration design, the tooth position, the restoration brand, the abutment nature, the cement and the occlusal parameters were crossed with the presence of veneer or framework fracture, using *t*-student and chi-square tests. The level of significance was set at $p < 0.05$. Data analysis was conducted first globally and then by type of restoration.

3. RESULTS

3.1 Clinical data about patients and reconstructions

During the inclusion period, 193 restorations were inserted in 65 patients. Among them, 39 were women. The mean age was 54.6 (SD 11.8) years with a range from 25 to 79 years at the time of examination. Data about the type of restoration, type of connection, brand, manufacturing process and mean follow- up are summarized in Table 3.3.

Type of restoration	Zirconia internal connection	Brand	Manufacturing process		Mean follow-up ± SD (months)
Abutments (n=46)	76.1% (n = 35)	Procera Etkon Straumann preshaped	Pre-sintered Pre-sintered Pre-sintered	50% (n = 22) 45.5% (n = 20) 4.5% (n = 2)	32.6 ± 25.4
Crowns - Cemented (n = 75)	-	Procera DC-Zirkon Lava	Pre-sintered HIP Pre-sintered	66.7% (n = 50) 24% (n = 32) 1.3% (n = 1)	49.7 ± 33.5
- Screw-retained (n = 30)	26.7% (n = 8)	Procera DC-Zirkon	Pre-sintered HIP	86.7% (n = 26) 13.3% (n = 4)	22.9 ± 19.0
FPDs - Cemented (n = 30)	-	Procera DC-Zirkon Cercon Lava	Pre-sintered HIP Pre-sintered Pre-sintered	56.7% (n = 17) 33.3% (n = 10) 6.7% (n = 2) 3.3% (n = 1)	47.6 ± 32.5
- Screw-retained (n = 12)	0% (n = 0)	Procera DC – Zirkon	Pre-sintered HIP	83.3% (n = 10) 16.6% (n = 2)	21.1 ± 12.0

Table 3.3 *Type of restoration, brand, manufacturing process and mean follow-up.*

Most single crowns (80%) were placed on premolars and molars. Among FPDs, 39.8% were anterior (related to incisors and/or canines), and 60.2% were posterior (related to premolars and/or molars). Three unit bridges were the most evaluated restorations (43.8%), while the largest FPDs reached 12 elements.

The cemented crowns and FPD abutments were on natural teeth (*n* = 38 and *n* = 21 respectively), or titanium abutments (*n* = 15 and *n* = 2), or zirconia abutments (*n* = 22 and *n* = 6). Only one anterior 3-unit FPD included both a natural tooth and a zirconia abutment.

The restorations were cemented either with a self-adhesive resin cement (Rely X Unicem (3M, ESPE; Seefeld, Germany) (49.3%) or Clearfil SA Cement (Kuraray, Sakazu, Okayama, Japan) (5.9%)), a self-etch resin cement (Panavia (Kuraray, Noritake Dental, Tokyo, Japan) (25%), or Clearfil aesthetic cement (Kuraray Medical, Okayama, Japan) (9.9%)), a resin-reinforced glass ionomer cement (Fuji+ (GC Europe, Leuven, Belgium) (5.9%)), or a temporary resin cement (Improv (Nobel Biocare, Gothenburg, Sweden) (3.9%)).

3.2 Occlusal risk factors

Data about the repartition of occlusal risk factors are detailed in Table 3.4.

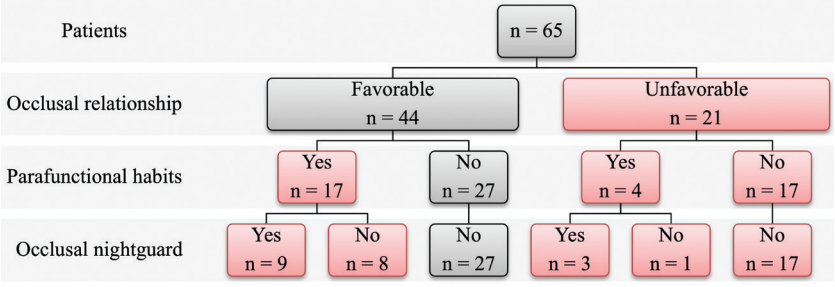

Table 3.4 *Repartition of occlusal risk factors.*

3.3 Restorations evaluation

After a mean observation period of 41.5 ± 31.8 months (range 6–107 months), the survival rate (which comprises all restorations still in the mouth even if failed) for 147 crowns and FPDs was 93.2% and the success rate (which takes

into account all failed restorations) was 81.6%. The estimated Kaplan-Meier survival rate was 91.2% at 9 years and the estimated success rate was 52.7% at 9 years.

3.3.1. Biological complications

Loss of vitality of two abutment teeth was found in two patients with an FPD after 2.7 and 23.9 months respectively. Root-canal treatment of the abutment teeth was successfully performed through an access cavity in the reconstructions. The endodontic treatment access holes were sealed with composite resin filling material. No secondary caries were found. After a mean observation period of 39.4 ± 30.5 months, the biological success and survival rates were 99% and 100%, respectively. No difference in probing pocket depth (PPD), probing attachment level (PAL), plaque control record (PCR), and bleeding on probing (BOP), of the test and control teeth respectively, was found in all types of ZBR.

3.3.2 Technical complications

3.3.2.1 Zirconia implant abutments

After a mean observation period of 32.6 ± 25.4 months, zirconia implant abutments (*n* = 46) showed a 100 % survival and success rate, whatever the type of connection.

3.3.2.2 Crowns and FPD's

USPHS rating of restorations is presented in Table 3.5. After a mean observation period of 41.5 ± 31.8 months (range 6–107 months), 25 restorations exhibited technical complications and the technical success rate was 83%. The technical survival rate was 93.2%: 10 restorations required replacement because of framework (*n* = 4) or veneer (D according to USPHS criteria) (*n* = 7) fractures. Fracture of the veneering ceramic was the most frequent complication as 22 restorations (15%) exhibited minor or major chipping. One-third of the patients affected by veneer fracture exhibited chipping both with ZBR and other types

of prostheses, such as reinforced lithium disilicate glass ceramic or PFM restorations. The technical evaluation by means of the USPHS criteria revealed no statistically significant differences between crowns and bridges. Occlusal wear, marginal adaptation and anatomical form were positively evaluated. No

	Type of restoration	Alfa (A)	Bravo (B)	Charlie (C)	Delta (D)
Framework fracture	Cemented crowns	100% (n = 74)	-	-	0% (n = 0)
	Screw-retained crowns	96.7% (n = 29)	-	-	3.3% (n = 1)
	Cemented FPDs	93.3% (n = 28)	-	-	6.7% (n = 2)
	Screw-retained FPDs	91.7% (n = 11)	-	-	8.3% (n = 1)
	Total	**97.3% (n = 142)**	**-**	**-**	**2.7% (n = 4)**
Veneering porcelain fracture	Cemented crowns	82.7% (n = 62)	6.7% (n = 5)	4% (n = 3)	6.7% (n = 5)
	Screw-retained crowns	93.3% (n = 28)	3.3% (n = 1)	3.3% (n = 1)	0% (n = 0)
	Cemented FPDs	86.7% (n = 26)	6.7% (n = 2)	6.7% (n = 2)	0% (n = 0)
	Screw-retained FPDs	75% (n = 9)	8.3% (n = 1)	0% (n = 0)	16.7% (n = 2)
	Total	**85% (n = 125)**	**6.1% (n = 9)**	**4.1% (n = 6)**	**4.8% (n = 7)**
Occlusal wear	Cemented crowns	97.3% (n = 73)	2.7% (n = 2)	0% (n = 0)	0% (n = 0)
	Screw-retained crowns	100% (n = 30)	0% (n = 0)	0% (n = 0)	0% (n = 0)
	Cemented FPDs	90% (n = 27)	10% (n = 3)	0% (n = 0)	0% (n = 0)
	Screw-retained FPDs	100% (n = 12)	0% (n = 0)	0% (n = 0)	0% (n = 0)
	Total	**96.6% (n = 142)**	**0% (n = 0)**	**0% (n = 0)**	**3.4% (n = 5)**

Table 3.5 *USPHS ratings of Y-TZP-based restorations.*

	Type of restoration	Alfa (A)	Bravo (B)	Charlie (C)	Delta (D)
Marginal adaptation	Cemented crowns	94.7% (n = 71)	5.3% (n = 4)	0% (n = 0)	0% (n = 0)
	Screw-retained crowns	100% (n = 30)	0% (n = 0)	0% (n = 0)	0% (n = 0)
	Cemented FPDs	86.7% (n = 26)	13.3% (n = 4)	0% (n = 0)	0% (n = 0)
	Screw-retained FPDs	100% (n = 12)	0% (n = 0)	0% (n = 0)	0% (n = 0)
	Total	**94.6% (n = 139)**	**5.4% (n = 8)**	**0% (n = 0)**	**0% (n = 0)**
Anatomical form	Cemented crowns	84% (n = 63)	16% (n = 12)	0% (n = 0)	0% (n = 0)
	Screw-retained crowns	93.3% (n = 28)	6.7% (n = 2)	0% (n = 0)	0% (n = 0)
	Cemented FPDs	96.7% (n = 29)	3.3% (n = 1)	0% (n = 0)	0% (n = 0)
	Screw-retained FPDs	83.3% (n = 10)	16.7% (n = 2)	0% (n = 0)	0% (n = 0)
	Total	**88.4% (n = 130)**	**11.6% (n = 17)**	**0% (n = 0)**	**0% (n = 0)**

Table 3.5 *Continuation.*

loss of retention was observed but one screw-retained restoration had to be screwed back after 3 months.

3.4 Fractographic analysis (Figures 3.1 and 3.2)

The veneer fractographic analysis was performed for 12 failed restorations out of 22, as some fractures had already been polished or the restorations replaced. Only one veneer fracture was considered as a delamination, which means that the zirconia framework was exposed. All veneer factures originated from occlusal roughness, except one, which initiated from the vestibular margin of the restoration.

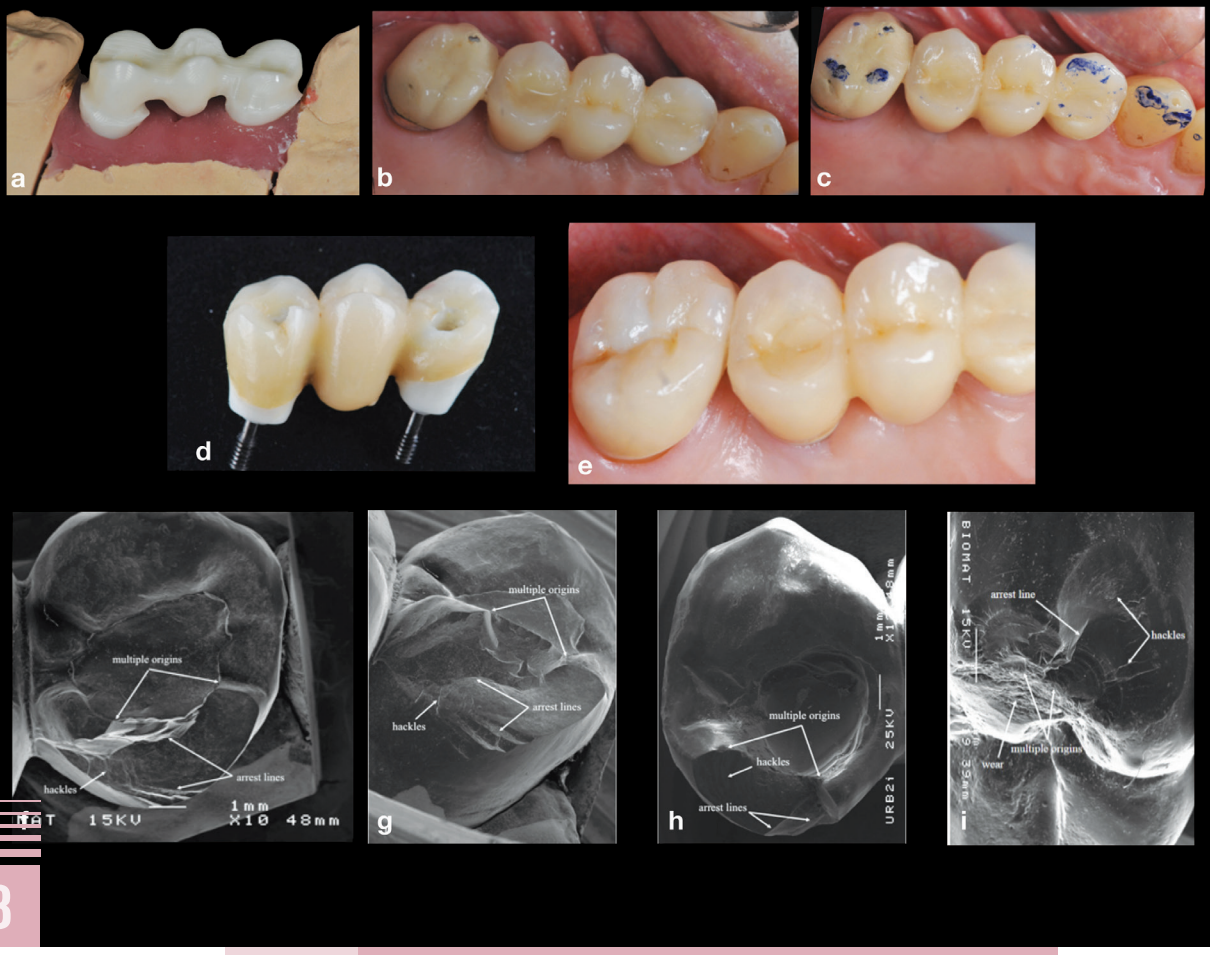

Fig. 3.1

(a) Three-unit screw-retained FPD with a supportive Y-TZP framework design.
(b) Bridge after placement. Note the PFM crown on tooth # 17, with a veneer fracture on the mesio-buccal surface.
(c) First major chipping on the palatal cusp of tooth # 14, 6 months after placement.
(d) FDP removed: major chipping (USPHS rating: D) is also visible on the palatal cusp of tooth # 16. This fracture occurred 27 months after restoration placement.
(e) Minor chipping (USPHS rating: B) on the mesio-buccal surface of the new crown on tooth # 17, as it had occurred in the previous PFM crown (Fig. 3.1b).
(f and g) Fractographic analysis of tooth # 14 replica, which revealed a complex multi-step veneer facture, which originated from the occlusal surface, close to the screw hole.
(h) Fractographic analysis of tooth # 16 replica, which revealed that the veneer facture originated from occlusal surface.
(i) Fractographic analysis of tooth # 17 replica, which revealed that the veneer facture originated from a rough area on the occlusal surface. (Photo courtesy of the author).

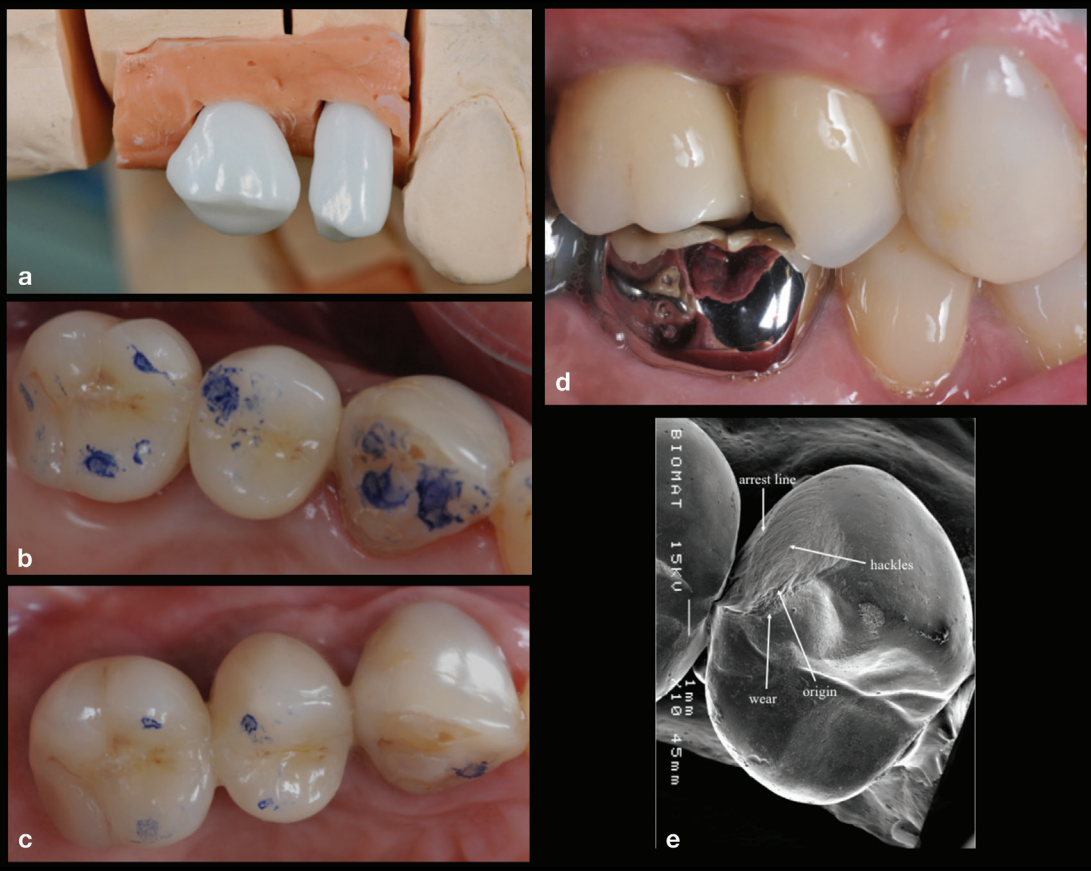

Fig. 3.2

(a) Y-TZP frameworks for cemented crowns on Y-TZP implant abutments on teeth # 15 and # 16.
(b) Minor chipping (USPHS rating: B) on the mesio-buccal surface of tooth # 16, which occurred 8 months after restoration placement.
Note the strong occlusal contact point on the occluso-distal surface of tooth # 15.
(c and d) Major chipping (USPHS rating: D) of the buccal cusp of tooth # 15, which occurred 9 months after restoration cementation.
(e) Fractographic analysis revealed that the crack origin was located on a rough area corresponding to the strong occlusal contact point with tooth # 46. (Photo courtesy of the author).

3.5 Correlation between fractures and clinical parameters

3.5.1 Veneer fractures

Veneer fractures were not related to a specific type of restoration (cemented, screw-retained, crown or FPD). The parameters, which were shown to be associated with chipping, were the nature of the antagonist (p = 0.013), the presence of parafunctional habits (p = 0.018) and the type of support (p = 0.026). More fractures were observed in implant-supported restorations with ceramic restorations as antagonist, in the presence of parafunctional habits. The use of an occlusal nightguard in patients with parafunctional habits significantly decreased the chipping occurrence (p = 0.0048). The brand significantly influenced the veneer fractures in cemented FPDs, (p = 0.0063): more fractures occurred with implant-supported restorations, with ceramic restorations as antagonists, with the brand DC-Zirkon. The number of elements in screw-retained FPDs was also related to chipping (p = 0.0036), long-span prosthesis showing more failures.

In most chipping cases (rating B or C according to USPHS criteria), the subsequent treatment was polishing. One screw-retained FPD (chipping rated D) was unscrewed and repaired with ceramic.

3.5.2 Framework fractures

Framework fractures were not related to a specific type of restoration (cemented, screw-retained, crown or FPD). Four framework fractures occurred: one 3-unit tooth-supported FPD, one 12-unit tooth-supported FPD with cantilevers at the end, one 12-unit screw-retained FPD and one fracture occurred on the internal implant connection of a screw-retained crown. Concerning FPDs, fractures always occurred on bridge connections. Overall, the clinical parameters, which were shown to influence framework fractures were the number of elements (p < 0.0001), the nature of the antagonist (p < 0.0001),

the type of support ($p < 0.0001$) and the brand ($p < 0.0001$). Indeed, more fractures were noted on Procera's long-span implant-supported restorations with ceramic restorations as antagonists.

3.6 Patient satisfaction level

The aesthetic outcome was rated 9.1 ± 1.1 (range 3–10) and the functional outcome was rated 8.8 ± 1.3 (range 3–10).
The patients presenting veneer fractures failures were shown to be significantly less satisfied than others ($p = 0.019$).

4. DISCUSSION

Zirconia abutments ($n = 46$) showed excellent results, with survival and success rates of 100% after a mean observation period of 32.6 ± 25.4 months, whatever the type of connection, the majority being internal. Similar results were found in the literature [47].

Regarding zirconia-based prostheses ($n = 147$), the survival rate was 93.2% after a mean observation period of 3.5 years. According to several studies, zirconia-based prostheses can achieve survival rates of 100% at 3 years [18,19,21,22,24,25,48–50] and even 5 years [8,9,10,15]. But these results are quite overestimated as most of these studies excluded patients with para-functional activity. In the present study, the success rate was 81.6% after a mean observation period of 3.5 years. The 9-year Kaplan-Meier estimated success rate was only 52.7%. As in previous studies [36], technical complications were the first cause of failures (17%). One screw-loosening was noted, but no restoration was unsealed. According to USPHS criteria, occlusal wear, marginal adaptation and anatomical form were evaluated favourably, confirming the results of

the literature [12,26,29,35,51]. Fracture was the first cause of failure. A 2.7% framework fracture rate (n = 4) was observed, which is also similar to other studies on ZBR [7,13,23], but is lower than studies on other ceramic material-based restorations [52,53]. Risk areas were the connection zones, which must be correctly dimensioned. However, this condition is not sufficient, as observed with a 12-unit tooth-supported FPD with distal cantilevers, which fractured through a large connection. Indeed, even if two studies [19,32] about posterior cantilever bridges showed high survival rates after 3 years, the use of zirconia in this indication is risky since the connections are submitted to high mechanical stress. It is the same with zirconia inlay-retained FPDs, which showed a 10% framework fracture rate at 1 year [34]. The results of the present study highlighted that long-span FPDs, implant-supported restorations, and the presence of ceramic restorations as antagonist promote framework fractures. Indeed, most of these parameters influence the amount of stress to which the framework is submitted. When comparing tooth- with implant-supported fixed dental prostheses, a systematic review highlighted that implant-supported exhibited more technical complications than tooth-supported restorations [54]. If implants provide a rigid support for the framework and reduce the bridge bending [55], they on the other hand reduce proprioception and then may increase the occlusal stress [35,49].

As expected, the fracture of the veneering ceramic was the main technical complication, with a 15% chipping rate, and the first cause of failure of zirconia-based prosthesis. This confirms the literature background, showing that the veneering ceramic is the weak link of ZBR [36]. As often reported, the majority of patients had not noticed these fractures, which had simply been polished [36]. However, seven restorations had to be replaced due to major chipping. The veneer fracture rates reported in the literature vary from one study to the other: from 0% at 2, 3 and 5 years [8,23,31], to 88.9% at 8 years [6]. The present results are slightly higher than the weighted (by sample size) average value of all studies (12.3%) (Table 3.1). However comparisons between studies are questionable since the diagnosis and the analysis of chipping failures are still not standardized, in

the absence of agreement some studies may not have taken into account very small chips [56]. Even if the actual tendency consists on the utilization of zirconia monolithic restorations, the veneering process is not out of date and the chipping resistance of ZBR needs to be improved. Indeed monolithic restorations have not proven yet their long-term clinical efficacy, as for example the absence of a veneering ceramic layer as a barrier for water penetration could be suspected to promote Y-TZP low thermal degradation, particularly in the occlusal surface, which is more submitted to wear process [57]. Moreover, from an aesthetic point view, zirconia monolithic restorations are less adapted to anterior restorations.

In the current work, the correlation between veneer fractures and some clinical parameters, which influence external stresses has been established. As for framework factures, the presence of a ceramic restoration as antagonist was a risk factor. Similarly, implant-supported restorations are riskier, probably due to the absence of stress resilience via the periodontal ligament [54]. These results are identical to anterior studies [12,35,49,54]. For screwed-retained restorations, the number of elements, and thus the amount of external stress to which the restoration is submitted, also promoted chipping. Furthermore, the present study revealed a significant association between the zirconia brand and the chipping rate. Indeed, on luted FPDs, a greater number of fractures was observed with the DC-Zirkon system, which consists of a hard-milling process of dense sintered Hot Isostatic Pressure (HIP) zirconia blocks. However, there is an antagonism between the occurrence of veneering ceramic fractures and framework fractures. Indeed, in this study, as in the literature [36], the pre-sintered (non-HIP) zirconia promoted significantly more framework fractures. This result can be explained by the lower density and then lower strength of pre-sintered in comparison with HIP zirconia.

The most important clinical risk factors highlighted were the presence of parafunctional activity and the absence of an occlusal nightguard. Similar results were reported in two previous studies [25,58]. The first study is a

retrospective evaluation of 94 implant-supported PFM FPDs in partially edentulous patients [58] and the second is a retrospective evaluation of 16 zirconia implant fixed complete dentures [25]. In these studies, the presence of parafunctional habits, the absence of an occlusal nightguard and implant-supported restorations as antagonists were proven to increase chipping. In the present study most fractures originated from occlusal surfaces, as confirmed by Sailer et al. [21], who also reported that chipping originated from roughness on occlusal contact points. Those results underline the influence of occlusal relationships on the mechanical behaviour. If masticatory loads are reported to be higher in the posterior than in the anterior region, this parameter had no significant impact on chipping in the present study, maybe because para-functional habits affect both areas. Since only one delamination was diagnosed, the zirconia-veneer adhesion does not seem to be the Achille's heel of the bilayer. The influence of manufacturing parameters cannot be excluded since for 5 out of 6 patients affected by several veneer fractures, the restorations were manufactured together. However the dental laboratory was not shown to have a significant influence on chipping occurrence. The influence of slow cooling procedures of the veneering ceramic, which were introduced recently by manufacturers to reduce failures, should be evaluated in future clinical studies. Finally this study showed that one-third of the patients affected by veneer fracture, exhibited chipping both with ZBR and other types of prostheses, such as reinforced lithium disilicate glass ceramic or PFM restorations.

This highlights the fact that, besides manufacturing and material choice, the patient and the clinical parameters are crucial influencing factors in the mechanical behaviour of dental prostheses.

5. CONCLUSIONS

In this study, ZBR showed a high survival rate (93.2%) after a mean observation period of 3.5 years. However, the success rate was 81.6% and the 9-year Kaplan-Meier estimated success rate was only 52.7%. The results confirmed that fracture of the veneering ceramic is the first cause of failure for ZBR, with a chipping rate of 15%.

Framework fractures, which always occurred on bridge connections, were related to prosthesis design, as the high number of elements or the presence of cantilevers, to the nature of the antagonist teeth, and to implant restorations. The analysis of veneer fractures support the influence of several clinical risk factors:

- the presence of parafunctional activity ($p = 0.018$) and the absence of an occlusal nightguard in these patients ($p = 0.0048$), as the most important factors;
- the presence of a ceramic restoration as an antagonist (0.013);
- the implant-supported restorations (0.026);
- for cemented FPDs: the dense HIP manufacturing systems ($p = 0.0063$);
- for screwed FPDs: the number of elements ($p = 0.0036$).

Fractographic analyses confirmed that the origins of fractures were almost always situated on the occlusal surfaces of the restorations.

The results of the present study underline the importance of external stress, i.e. functional stress, in regards to chipping occurrence. This stress adds to residual stress developed during the manufacturing process, but is a patient- and not a material-related parameter, which contributes to the explanation of a complex mechanism.

The association between chipping and clinical risk factors, particularly occlusal parameters, should be considered in future prospective clinical studies. Dental practitioners should pay specific attention to clinical parameters when performing ZBR until a solution is found to improve the mechanical resistance of the materials.

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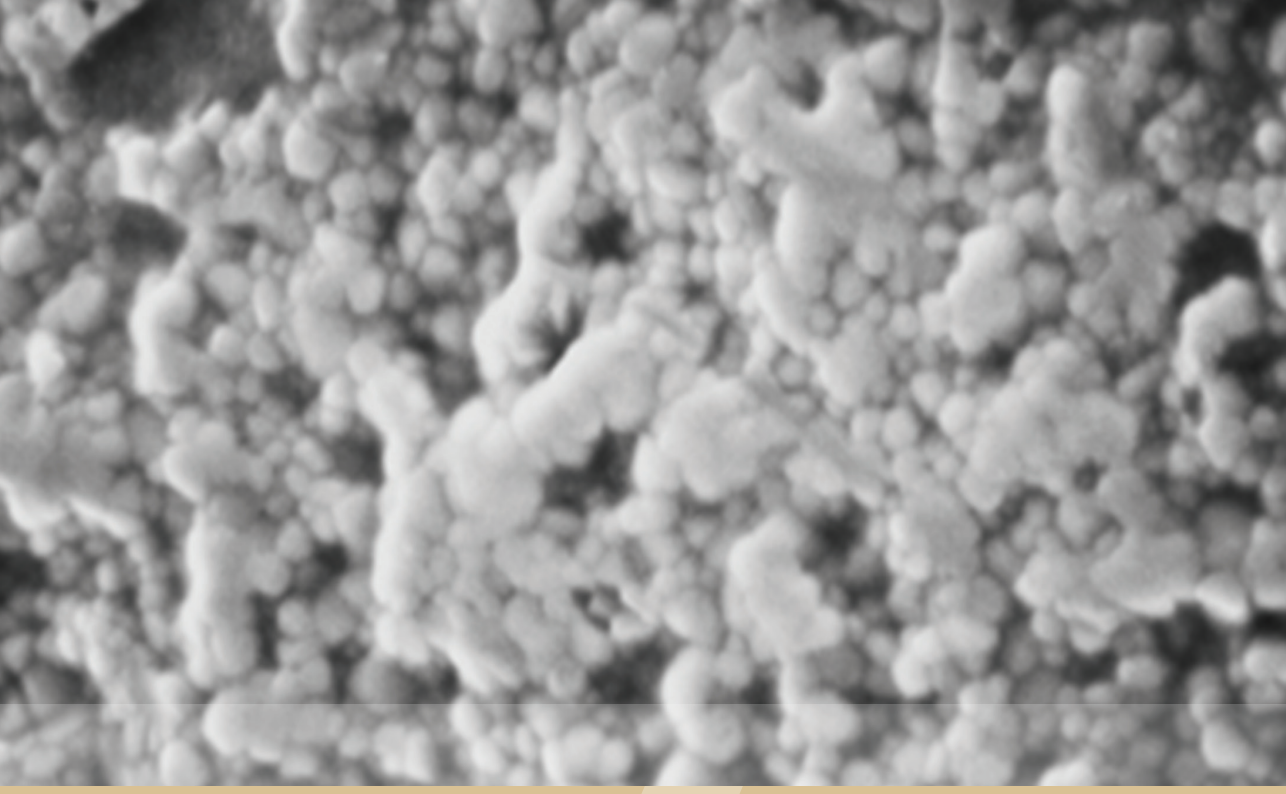

4

Aging of monolithic zirconia dental prostheses: Protocol for a 5-year prospective clinical study using *ex vivo* analyses

Vinciane Koenig [1]
Claudine P. Wulfman [2, 4]
Mathieu A. Derbanne [2]
Nathalie M. Dupont [1]
Stéphane O. Le Goff [2]
Mie-Leng Tang [2]
Laurence Seidel [3]
Thibaut Y. Dewael [1]
Alain J. Vanheusden [1]
Amélie K. Mainjot [1, 2]

1 *Dental Biomaterials Research Unit (d-BRU) and Department of Fixed Prosthodontics, Institute of Dentistry, University of Liège (ULg) and University of Liège Hospital (CHU), 45 Quai G. Kurth, Liège, 4020, Belgium.*

2 *Unité de Recherches en Biomatériaux Innovants et Interfaces (URB2i) – EA4462, Faculté de Chirurgie Dentaire, Université Paris Descartes, Sorbonne Paris-Cité, Montrouge, 92120, France.*

3 *Biostatistics Department, University Hospital of Liège, Belgium*

4 *Service d'odontologie, Hôpital Louis Mourier, Assistance Publique-Hôpitaux de Paris (URB2i), France*

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ABSTRACT

Background: Recent introduction of computer-aided design/computer-aided manufacturing (CAD/CAM) monolithic zirconia dental prostheses raises the issue of material low thermal degradation (LTD), a well-known problem with zirconia hip prostheses. This phenomenon could be accentuated by masticatory mechanical stress. Until now zirconia LTD process has only been studied *in vitro*. This work introduces an original protocol to evaluate LTD process of monolithic zirconia prostheses in the oral environment and to study their general clinical behavior, notably in terms of wear.

Methods/design: 101 posterior monolithic zirconia tooth elements (molars and premolars) are included in a 5-year prospective clinical trial. On each element, several areas between 1 and 2 mm^2 (6 on molars, 4 on premolars) are determined on restoration surface: areas submitted or non-submitted to mastication mechanical stress, glazed or non-glazed. Before prosthesis placement, *ex vivo* analyses regarding LTD and wear are performed using Raman spectroscopy, SEM imagery and 3D laser profilometry. After placement, restorations are clinically evaluated following criteria of the World Dental Federation (FDI), complemented by the analysis of fracture clinical risk factors. Two independent examiners perform the evaluations. Clinical evaluation and *ex vivo* analyses are carried out after 6 months and then each year for up to 5 years.

Discussion: For clinicians and patients, the results of this trial will justify the use of monolithic zirconia restorations in dental practice. For researchers, the originality of a clinical study including *ex vivo* analyses of material aging will provide important data regarding zirconia properties.

Trial registration: ClinicalTrials.gov Identifier: NCT02150226.

Keywords: Dental prosthesis, Zirconia, Low thermal degradation, Computer-aided design/computer-aided manufacturing, Wear, Raman spectroscopy

1. BACKGROUND

Dental caries and periodontal diseases affect nearly 100% of the adults worldwide [1,2]. Crowns are intended to restore a tooth with extensive decay, while bridges are intended to replace at least one missing tooth. Crowns and bridges can also be used on dental implants. Thanks to the emergence of computer-aided design/computer-aided manufacturing (CAD/CAM) processes, zirconia (yttria-tetragonal zirconia-polycrystal, Y-TZP), a polycrystalline ceramic material, was introduced to replace metal in dental prostheses because of its good mechanical, better optical properties and good biocompatibility. These prostheses are typically bilayered structures, with a framework that gives mechanical resistance and a porcelain layer that provides aesthetics to the restoration. Unfortunately, clinical reports on zirconia-based restorations have indicated a high rate of short-term failures related to cohesive fracture of the porcelain layer [3], which constitutes a weak link from a mechanical point of view. Therefore, manufacturers have recently introduced monolithic prostheses, which are fully composed of zirconia, without any porcelain layer, except for a thin layer of glaze.

Currently, few clinical studies have been published on zirconia monolithic restorations [4-12]. Yet a critical issue with those restorations is the material low thermal degradation (LTD), which generates zirconia surface degradation, loss of mechanical properties and risk of fracture [13-17]. Indeed, zirconia LTD is an aging phenomenon occurring when the material is in contact with water, which induces a change in zirconia metastable crystalline structure. LTD was intensely investigated in the orthopaedic field following numerous zirconia hip prosthesis fractures encountered in the 2000's [18]. Consequently, several *in vitro* studies were performed concerning LTD of dental prostheses [13,14,19-24]. Most particularly, LTD was shown to be responsible for a decrease in material flexural strength when 50% of sample surface crystals are transformed [21,25,26]. For zirconia dental implants, International Standard Rules [27,28] state that the crystalline transformation must not

exceed a maximum of 25% after aging in an autoclave at 134°C, 2 bar for 5 h, while no guidelines are available for zirconia prostheses. Nonetheless, extrapolation of *in vitro* results to clinical behavior is debatable with respect to the differences between oral environment and autoclave aging. Moreover, *in vitro* studies did not take into account the effect of mastication mechanical stress on restorations [26,29-31]. Consequently, the prediction of LTD kinetics and its impact on the lifespan of dental prostheses remains an unsolved problem. To author's knowledge, no clinical studies about *in vivo* LTD of dental zirconia prostheses has been published up to now. This issue is particularly critical for monolithic zirconia restorations that have no porcelain layer to act as a barrier against water penetration [31,32] and which can be submitted to glaze wear. Additionally, some high translucency Y-TZP developed for monolithic restorations are reputed to be more metastable and, thus, more sensitive to LTD [33].

2. AIMS AND OBJECTIVES

The main objective of this 5-year prospective study is to evaluate the *in vivo* LTD of monolithic zirconia restorations on implants and natural teeth using an original protocol, which includes *ex vivo* analyses of zirconia crystalline microstructure. Secondary objectives include the investigation of the overall quality of monolithic restorations and of the wear process effect on both restorations and antagonistic teeth. The glaze LTD protective effect is investigated through a comparison of glazed and unglazed areas, submitted or not to mastication mechanical stress.

3. DESIGN AND METHODS

3.1. Study design

A 5-year prospective trial was designed. It received approval from the Ethics Committee of the University of Liège (Comite d'Ethique Hospitalo-Facultaire Universitaire de Liège, number B7107201317778, reference 2013/138). Table 4.1 gives an overview of the study, which is composed of three stages: zirconia prostheses realisation, baseline data gathering and follow-up evaluations (after 6 months and every year for up to 5 years). Evaluations include clinical evaluation and *ex vivo* analyses.

3.2. Participants and settings

3.2.1 Settings

Patients are included and treated in the Department of Fixed Prosthodontics, Institute of Dentistry, University Hospital, Liège, Belgium. Any patient with the eligible criteria visiting the Institute of Dentistry is asked to participate in the study.

3.2.2 Inclusion/exclusion criteria

Patients are eligible to participate in the trial if they need restoration(s) in the posterior region (molar or premolar). The restorations can be carried out either on implants or teeth. Multi-unit restorations on implants are included if limited to 3 elements (maximum 2 bridges per patient). Several teeth per patient are eligible (maximum 6 elements per patient).

Patients presenting parafunctions such as bruxism, masticatory muscle discomfort, articular disorders or severe wear facets were also included. Exclusion criteria are severe and acute periodontal, carious disease or poor

oral hygiene. Patients with removable prosthesis as an antagonist are excluded. Once eligibility is established, the protocol is presented and explained to patients. Inclusion is validated after consent signature.

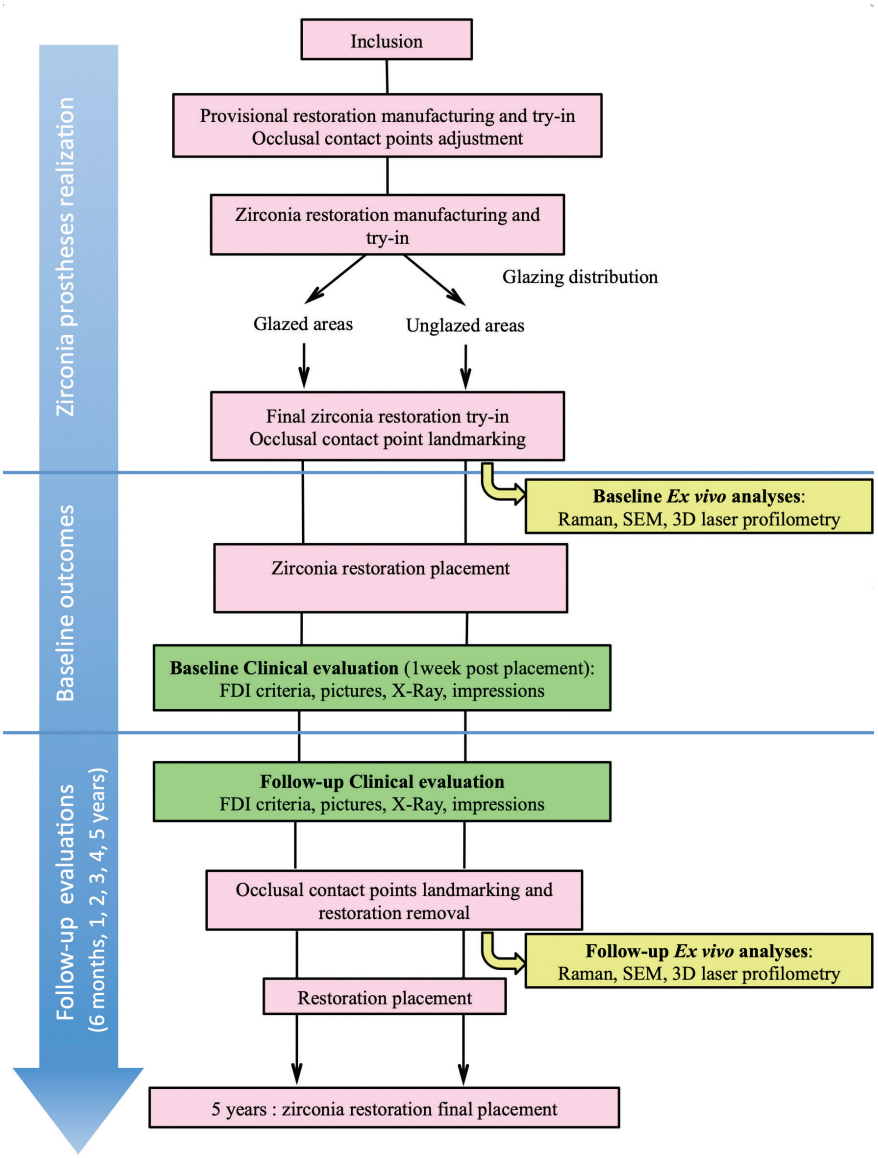

Table 4.1 *Study design.*

3.2.3 Operators and evaluators standardization

Operators carry out prosthetic treatment. Evaluators assign scores according to FDI criteria. Both operators and evaluators are experienced dentists in the field of fixed prosthodontics. They are trained in the FDI criteria by means of the e-calib web based software (http://zep01793.dent.med.uni-muenchen.de/moodle/ website) and group training sessions. Operators cannot evaluate their own treatments. Trained researchers and technicians perform *ex vivo* analyses.

3.2.4 Participant incentives

Participants receive no financial compensation. However, their treatment and prostheses are provided free of charge. If the patient wishes to withdraw from the study, a conventional crown will be made at his expense. If an experimental crown fails during the study, a conventional crown will be provided as a replacement.

3.3. Procedure

3.3.1 Tooth preparation and impression for tooth or implant-supported prostheses

All clinical and technical procedures are performed in strict agreement with the clinical and technical instruction protocol validated by the ethics committee and following manufacturer's recommendations. Teeth are prepared following standardized criteria (1.0-1.5 mm occlusal depth cut to achieve appropriate occlusal anatomy, 1.0-1.5 mm functional cusp tip reduction, 0.5 mm gingival chamfer reduction, and a 6-8° taper to the axial walls). A double-mix impression is performed with a high- and a low-viscous A-silicone impression material (Aquasil Heavy/XLV, Dentsply De Trey, Konstanz, Germany) and the same

impression procedure is used for implant restorations. Shade is registered using Vita Classic System (Vita Zahnfabrik, Bad Säckingen, Germany) and if needed, restorations on antagonistic teeth are replaced.

3.3.2 Provisional restoration

Before the manufacture of zirconia restorations, CAD-CAM composite provisional crowns (Lava Ultimate, 3M ESPE, Seefeld, Germany) or PMMA provisional bridges are made. After die scanning, the restoration design is carried out with CAD/CAM software, either Exocad (Darmstadt, Germany) or Dental Wings (Montreal, Canada) (DPI Lava milling center, Anderlecht, Belgium). Specific buccal and palatal grips are added to the crown design to facilitate cemented crown removal. The file is then transferred to the milling machine for manufacturing (Lava CNC 500, Serial Number: 07019 (2009), 3M ESPE).

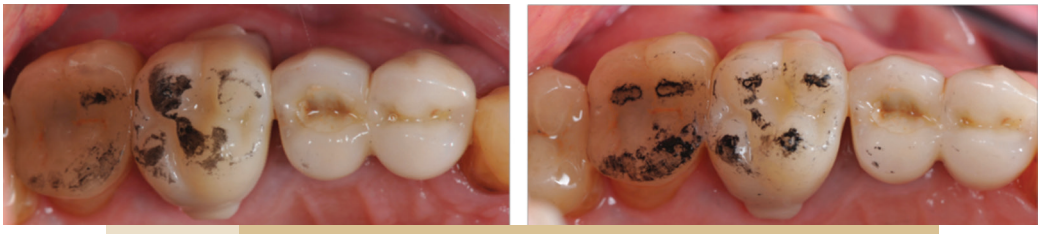

Fig 4.1 *Occlusal contact points before and after adjustment on a Lava Ultimate crown (tooth #16).*

The provisional restorations are adapted in-mouth and used as a template for the design of the zirconia restoration. Particular attention is paid to occlusal contact points adjustment, in order to obtain at least one flat contact surface of approximately 1 mm^2 per cusp, by either grinding or by adding composite (Fig. 4.1).

3.3.3. Zirconia prostheses

Provisional restorations are scanned for zirconia restorations fabrication (Lava Plus, 3M ESPE, Seefeld, Germany) with the same milling system. Sintering

is performed according to manufacturer's instructions, i.e. at 1450 °C for 2 h. Implant-supported restorations are bonded on to a specific titanium abutment (1000er-Serie, Medentika, Hugelsheim, Germany) with a resin composite cement: either RelyX Ultimate (3M ESPE, Seefeld, Germany) for the first 16 restorations of the study, or Multilink abutment (Ivoclar Vivadent, Schaan, Liechtenstein) for the 40 next, according to manufacturer's recommendations, after sandblasting of the abutment and of the zirconia restoration with 50 µm alumina particles, 2 bar. Zirconia restorations are tried-in and occlusal contact points are adjusted and polished with a specific bur kit if needed (Diasynt Plus/Diacera Zirconium, Eve Ernst Vetter, Pforzheim, Germany). Adjusted areas are encoded.

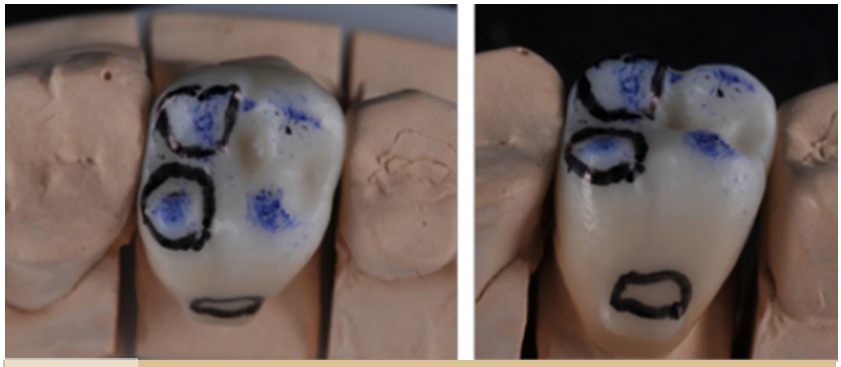

Fig 4.2 *Landmarking with permanent ink of areas, which will not be glazed (tooth #16).*

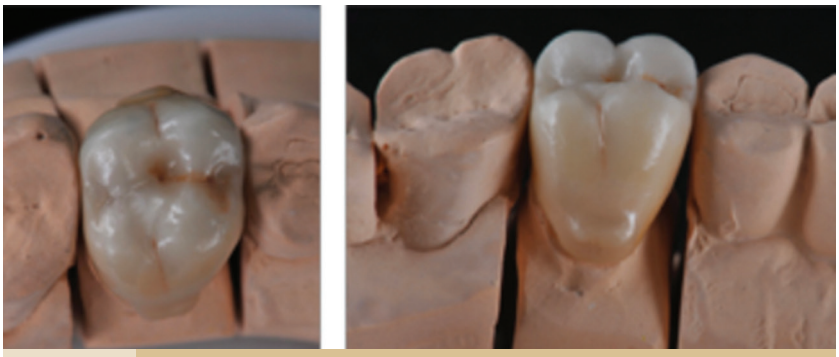

Fig 4.3 *Glazed Lava Plus crown (tooth #16).*

Occlusal surface contact areas, which will not be glazed, are randomly determined (Figs. 4.2 and 4.3). Four occlusal contact points (one contact per cusp) are determined on molars and two on premolars. For molars, two cusps are randomly selected to remain unglazed: one centric cusp (unglazed centric cusp (UCC)) and one non-centric (unglazed non-centric cusp (UNCC)). The two other cusps are called "glazed centric cusp" (GCC) and "glazed non-centric cusp" (GNCC). For premolars, one cusp is randomly selected to remain unglazed. Control areas are the buccal face (glazed) and the lingual/palatal face (unglazed) of the restoration. The glaze (IPS empress stains and eMax Ceram glaze, Ivoclar Vivadent, Schaan, Liechtenstein) is sintered at 780 °C for 1 min. Definitive bonding (bond is eliminated during the glaze firing) on the specific titanium abutment is performed following the procedure described previously. The glazed restorations are tried-in and occlusal contact points, as well as lingual/palatal and buccal areas, are marked for *ex vivo* analyses and registered with a picture (Fig. 4.4).

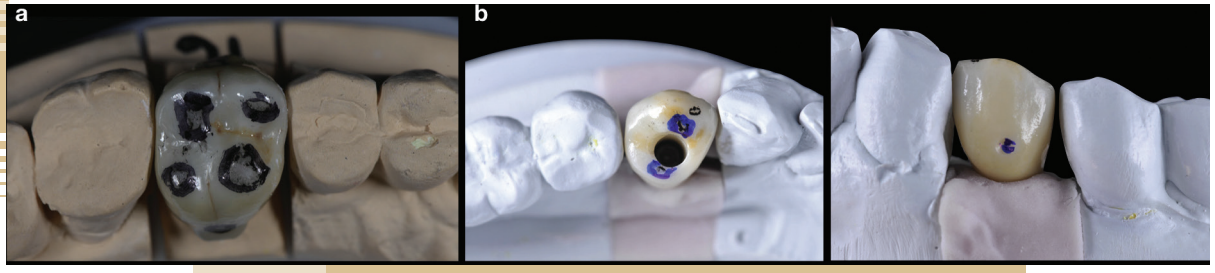

Fig 4.4 *Glazed crowns after try-in and landmarking of areas to be ex vivo analysed*
a) Final crown on tooth #16. Landmarking of areas to be analysed: occlusal contact points and control areas on buccal and palatal faces, which are located up to the undercut created to remove the crown.
b) Screw-retained crown on implant (tooth #34). Landmarking of areas to be analysed: occlusal contact points and control areas on buccal and lingual faces, which are located up to a small groove performed in the restoration surface.

3.3.4 Zirconia prostheses placement and removal

Baseline *ex vivo* analyses of zirconia restorations are performed before placement. Screw-retained restorations are torqued with 35 Ncm^{-1} (Fig. 4.5). Cemented restorations are sealed with eugenol-free cement (RelyX Temp NE,

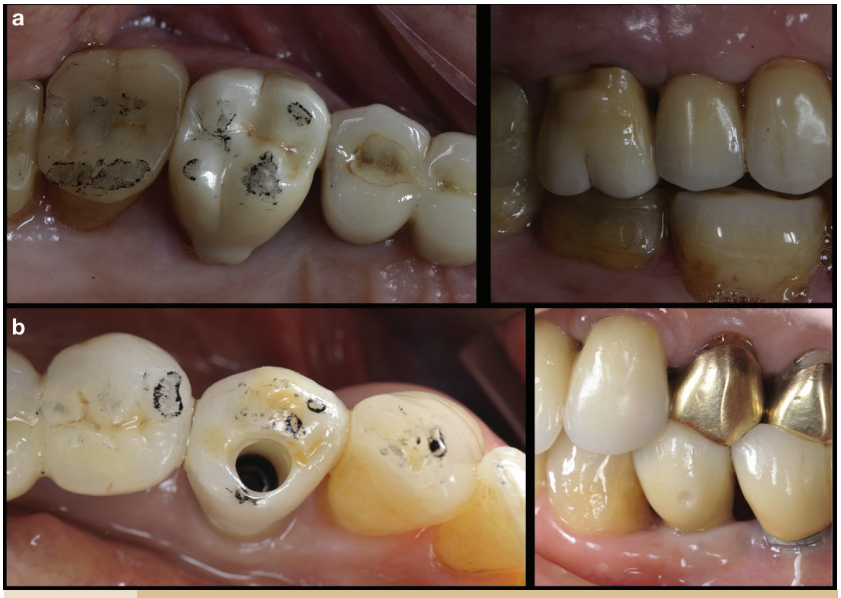

Fig 4.5 *Crowns after placement*
a) Cemented crown on tooth #16
b) Implant-supported crown on tooth #34.

3M ESPE) and prior to cementation, restorations are cleaned with alcohol in an ultrasonic bath and teeth are disinfected with 2% chlorhexidine. Clinical evaluation is performed one week after placement. After 6 months, restorations are clinically evaluated and then removed for *ex vivo* analyses. Provisional restorations replace zirconia restorations during *ex vivo* analyses. After these analyses, zirconia restorations are placed in the mouth of the patient, following the same procedure as the first time. Evaluations will be repeated after a one-year in-mouth stay, and then each year for up to 5 years.

3.4. Data collection

3.4.1 Primary outcome: LTD evaluation

LTD is evaluated directly on zirconia restorations through zirconia crystalline microstructure analysis with Raman spectroscopy. Indeed, LTD is characterized

by a shift from the tetragonal crystalline form (*t*) to the monoclinic form (*m*). The presence of monoclinic, tetragonal or a combination of both forms is distinguishable and quantifiable on Raman spectra, allowing the measurement of the transformation volume ratio (V_{fm}).

Raman spectra are recorded with a Labram Raman spectrometer (Horiba-Jobin Yvon, Kyoto, Japan). The excitation laser is provided by a HeNe laser (632 nm) with 1 mW power focused at the surface of the specimen and the Raman spectra are acquired by a charge-coupled device detector (Horiba-Jobin Yvon, Kyoto, Japan) with 1 cm^{-1} spectral resolution (1800 grooves/mm grating). The Raman spectrometer is combined with an optical microscope (Olympus LX71; Olympus Corporation, Tokyo, Japan). A confocal pinhole with adjustable diameter is used for a confocal detection and an objective 80x (numerical apertures 0.75) is used to reach 1 μm^3 resolution (lateral x axial).

Analysis of collected spectra enables V_{fm} calculation in the confocal probed volume, estimated using the Eq. (1) **[34]**:

$$V_{fm} = \frac{I_m^{178} + I_m^{189}}{0.33(I_t^{145} + I_t^{256}) + I_m^{178} + I_m^{189}}$$

where I_m and I_t are the intensities of the peaks (wave numbers in superscript) of the monoclinic and tetragonal phases. The Raman peak positions and intensities are obtained by fitting the Raman spectra with Lorentzian curves (Origin 8 software, OriginLab, Northampton, MA). 5 points per area are investigated and the outcome is the highest (worst) V_{fm} (%) for each area and tooth.

3.4.2 Secondary outcomes

3.4.2.1 Clinical evaluation

Clinical evaluation follows World Dental Federation recommendations and uses World Dental Federation instruments for assessing dental restorations,

described in 2007 [35] and updated in 2010 [36]. This instrument contains three dimensions (18 items): biological (six items), functional (seven items) and aesthetic (five items). Each item is assessed by clinical examination on a 5-point Likert scale (1 corresponding to a perfect restoration and 5 corresponding to a restoration that needs to be replaced) and collected. The dentist assesses all items except one; the remaining item is the patient-reported satisfaction. The outcome is the worst score of all items (ranging from 1 to 5) at follow-up. These evaluations are performed at baseline, at 6 months and then each year for up to 5 years by two independent evaluators. Moreover, occlusal risk factors are registered [3]: occlusal relationships characterized as favourable or unfavourable based on the clinical examination (class III or class II.2 malocclusion, anterior or posterior crossbite, edge to edge or open bite, were considered as unfavourable occlusal relationships), the presence of parafunctional habits, the use of an occlusal nightguard, the type of support (tooth or implant) and the nature of the antagonistic tooth. Impressions of restorations and antagonistic teeth are performed in order to cast polyurethane replicas (Alphadie, Schütz Dental GmbH, Rosbach, Germany). Beside radiographs, pictures of restorations and antagonistic teeth, with occlusal contact point registering, are performed. To prepare *ex vivo* analyses, occlusal contact points, as well as lingual/palatal and buccal areas are marked with permanent ink.

3.4.2.2 Wear

Wear is studied with *ex vivo* analyses of zirconia restorations, which include scanning electron microscopy (SEM) and 3D laser profilometry. Polyurethane replicas of teeth will be used to study wear of antagonistic teeth in the same manner, while replicas of zirconia restorations are stored as a control.

3.4.2.3 SEM observations

After Raman spectroscopy, restorations are gold-coated and observed with a JSM-6400 Scanning Electron Microscope (JEOL Limited, Tokyo, Japan). Interpretation of fracture patterns, if occurs, is based on the descriptions by Scherrer et al. [37], particularly to determine the origin and direction of the crack propagation.

3.4.2.4 3D laser profilometry

Samples are placed in the scanner on a die replica embedded in resin, for repeatable positioning at each evaluation. Occlusal, buccal and lingual surfaces are scanned with a custom-made device including a XY motorized board stage and a 100 nm-resolution laser sensor (Keyence LK G30 with LK GD500 controller, Keyence Corporation, Osaka, Japan). Raw data acquisition and processing are performed using a custom-developed software using C# language (Microsoft Visual Studio 2013, Microsoft Corporation, Redmond, WA, USA/Measurement Studio 2014, National Instrument Corporation, Austin, TX, USA) coupled to a digital data acquisition PCI board (NI PCI-6534, National Instruments Corporation, Austin, TX, USA). Resulting matrices of Z values are then transferred to a surface matching software Geomagic Control 2014 (Geomagic Inc, Morrisville, C.C., USA).

3.4.3 Data management

Data are collected, stored and processed in the Department of Fixed Prosthodontics, Institute of Dentistry, University Hospital, Liège, Belgium. Patients are identified by their inclusion number in order to preserve their privacy. Data are entered twice by operators and checked by a data manager. Only the data manager and statisticians have unrestricted access. Adverse events are also assessed at each study visit.

4. STATISTICAL ANALYSIS

4.1 Sample size

The determination of the sample size (N) was based on the following considerations. The statistical unit was the tooth characterized by its maximum LTD value recorded at each time point (baseline, 6 months, 1, 2, 3, 4

and 5 years). An LTD value above 50% was considered as treatment failure for the tooth. The overall proportion (π) of such treatment failures was defined as the primary outcome measure of the study. The study rationale was to reject the proposed treatment if $\pi > 0.20$, i.e. more than 20% treatment failures over time. Assuming a significance level α of 1% (Bonferroni correction for multiple time testing), a power 1-β of 90%, a proportion π of at most 0.08 (margin 0.12) and a one-sided Z test for a Binomial proportion of 0.20, a sample of 91 teeth would be needed to detect a percentage > 20% of treatment failures at each data point collection. To account for correlations between teeth within subjects and for study withdrawals, the sample size was increased to a minimum of $N = 100$ teeth.

4.2 Statistical methods

Quantitative variables characterizing patients and teeth are summarized by mean and standard deviation (SD) or by median and interquartile range (IQR) for skewed data; frequency tables are used for categorical variables. The association between two quantitative variables is assessed by the correlation coefficient. Cohen kappa coefficient is used to assess the degree of agreement between clinical evaluations made by different evaluators. The observed percentage of treatment failures at each time point (interim analysis) is tested at the 1% critical level by a one-sided Z test for a Binomial proportion of 0.20 as described in the sample size section. In case of rejection, the study will be terminated unless prostheses are not fractured and still functional in which case it will go on to analyze the LTD kinetic process. To assess the effect of fixed experimental factors (e.g. time, glaze, mechanical stress) and random effects (subjects and teeth) on LTD, wear measures and other clinical parameters, a generalized linear mixed model approach is used. Unless otherwise stated, results are considered significant at the 5% critical level. All calculations will be performed with the SAS (version 9.4) statistical package.

5. DISCUSSION

CAD-CAM processes have revolutionized the world of dental prostheses and the replacement of artisanal work by industrial processes has enhanced the reproducibility and the productivity of manufacturing. But one of the main advantages of CAD-CAM processes is the opportunity to use high performance materials, such as zirconia, particularly yttria-tetragonal zirconia-polycrystal (YTZP), a popular material, which was introduced in the early 2000's as an alternative to metal for crowns and bridges. Zirconia has good optical and biocompatibility properties in comparison with metal alloys and it is also the most resistant material among dental ceramics, combining high strength and toughness due to its unique phase transformation toughening property. Indeed, Y-TZP is a polycrystalline ceramic material in a metastable state: yttrium oxide acts as a dopant to stabilize the crystalline tetragonal form at room temperature, this tetragonal form being able to further transform to the monoclinic form under the effect of stress. This transformation is characterized by a crystal volume increase, which is able to counteract the propagation of cracks **[38]**. Unfortunately, this phase transformation can also occur with time, when the material is in contact with water, which is able to penetrate the crystalline structure. This aging phenomenon, called the low temperature degradation (LTD), generates zirconia surface degradation, loss of mechanical properties and risk of fracture **[13-17]**. LTD was at the origin of catastrophic failures encountered with zirconia hip prostheses in the early 2000's. This problem was extensively studied *in vitro*, particularly by Chevalier et al. **[18]**, but surprisingly, this issue was not raised by the dental community before the introduction of zirconia prostheses to the dental market. Yet temperature, moisture and mastication mechanical stress characterizing the oral environment are ideal conditions for LTD to develop and to impact the prognosis of dental prostheses. This is particularly true for monolithic zirconia restorations that are not covered by a porcelain layer preventing water penetration **[31,32]** and that are, for aesthetic reasons,

composed of specific high translucency varieties of zirconia, which can be particularly LTD-sensitive. Indeed, to increase translucency, some manufacturers increase grain size or reduce dopant content, which give more metastable zirconia [33].

Consequently, the primary outcome of this study protocol is to evaluate the in-mouth LTD of monolithic zirconia restorations on natural teeth and implants. Indeed, if, as suspected, LTD occurs in the oral environment, the question is the kinetics of this process and its impact compared to the lifespan of dental prostheses (around 15 years). To the author's knowledge, no clinical study about LTD of dental zirconia prostheses has been published up to now and the clinical background with monolithic restorations is too short to highlight potential failures. However, several *in vitro* studies were dedicated to this issue using artificial aging with an autoclave [13,14,19-24]. A recent systematic review [26] concluded that aging in an autoclave promotes Y-TZP LTD, decreases its flexural strength, while the monoclinic content increases. When increasing time (more than 20 h), pressure (more than 2 bars) and temperature (134 °C), the flexural strength significantly decreases, which was observed when the monoclinic content was superior to 50% in the sample surface. It must be noticed that none *in vitro* studies took into account the additional effect of mechanical stress on LTD [26,29-31]. Some authors showed a lower resistance to LTD for some high translucency zirconia than for standard zirconia, with the presence of around 75% of monoclinic content after 200 h of autoclave aging [39] and a decrease of 30% in crown resistance to cyclic mechanical loading after 100 h aging [40]. It must also be noted that only 1 h of exposure in a steam vapor autoclave at 134 °C and 2 bar is considered to correspond to 3 or 4 years of clinical use [41]. Yet extrapolation of *in vitro* aging to clinical behavior is doubtful, notably in regards to the important differences between oral environment and autoclave conditions, such as the absence of mechanical stress. If International Standard Rules [27,28] established for zirconia dental implants (not prostheses) state that the crystalline transformation must not exceed a maximum of 25% after aging in an

autoclave at 134 °C, 2 bar for 5 h, there are no guidelines regarding Y-TZP dental prostheses. Consequently, the present protocol, which combines clinical evaluation and *ex vivo* analyses, was designed to allow the monitoring of LTD in the oral environment through quantification of zirconia *t-m* phase transformation with Raman spectroscopy. Raman spectroscopy is a powerful and reliable method, which is an alternative to X-ray diffraction [42,43]. Its advantage lies in its 1 μm^2-resolution, which is particularly appropriate for the evaluation of occlusal contact points.

Regarding secondary outcomes of this study protocol, they include the investigation of the overall quality of monolithic restorations and of wear of both restorations and antagonistic teeth. Few clinical studies have been published in the literature concerning monolithic zirconia restorations and the clinical background is short [4-12]. Three studies focused on the evaluation of zirconia crowns and antagonistic teeth wear. They all used impressions and casting of replicas for an indirect quantification of the wear by 3D surface laser analysis, which can generate some bias related to the accuracy of replicas. The *ex vivo* analyses performed in the present protocol are intended to avoid this bias. Moreover, a supplementary advantage of *ex vivo* analyses is the direct observation of restoration with SEM, which allows the visual detection of glaze wear. As glaze wear could promote LTD, glaze protective effect is investigated through a comparison of glazed and unglazed areas, submitted or not to mastication mechanical stress, to evaluate the effect of this stress on LTD. Additionally, the general clinical behavior of monolithic zirconia tooth- and implant-supported restorations is seriously evaluated taking into account international standard criteria complemented by the analysis of a variety of risk factors, particularly occlusal, that can significantly influence the performance of the restorations, notably in terms of wear or fracture [3].

In conclusion, this new clinical protocol including in-depth *ex vivo* evaluation of Y-TZP microstructure will provide important data regarding its phase transformation process, which is still not fully understood, particularly in regards

to the effect of the combination of mechanical stress to moisture and temperature [32]. The novel approach of restoration removal at the different evaluation times allows for the use of Raman spectroscopy, SEM imagery and 3D laser profilometry to provide quantitative and qualitative information about Y-TZP aging and degradation of monolithic restorations. For future research, this trial should be able to provide reliable data to compute *in silico* models of dental zirconia in-mouth aging kinetics [41,44]. Indeed, there is an urgent and crucial need to establish standards regarding LTD of zirconia materials for dental prostheses on an international level in order to avoid potential failures in these restorations, used daily in dental offices.

TRIAL STATUS

The trial was submitted for registration at ClinicalTrials.gov on May 26, 2014. Patient recruitment started on February 2014. This protocol was submitted for publication on March 7, 2016.

COMPETING INTERESTS

The authors declare that they have no competing interests.

AUTHOR'S CONTRIBUTIONS

V. Koenig, C. P. Wulfman, M.A. Derbanne and A.K. Mainjot contributed to conception and design, data analysis and interpretation, drafted the manuscript. N.M. Dupont, S.O. Le Goff, M-L. Tang, L.Seidel, T.Y. Dewael, A.J. Vanheusden contributed to data analysis and interpretation, critically revised the manuscript.

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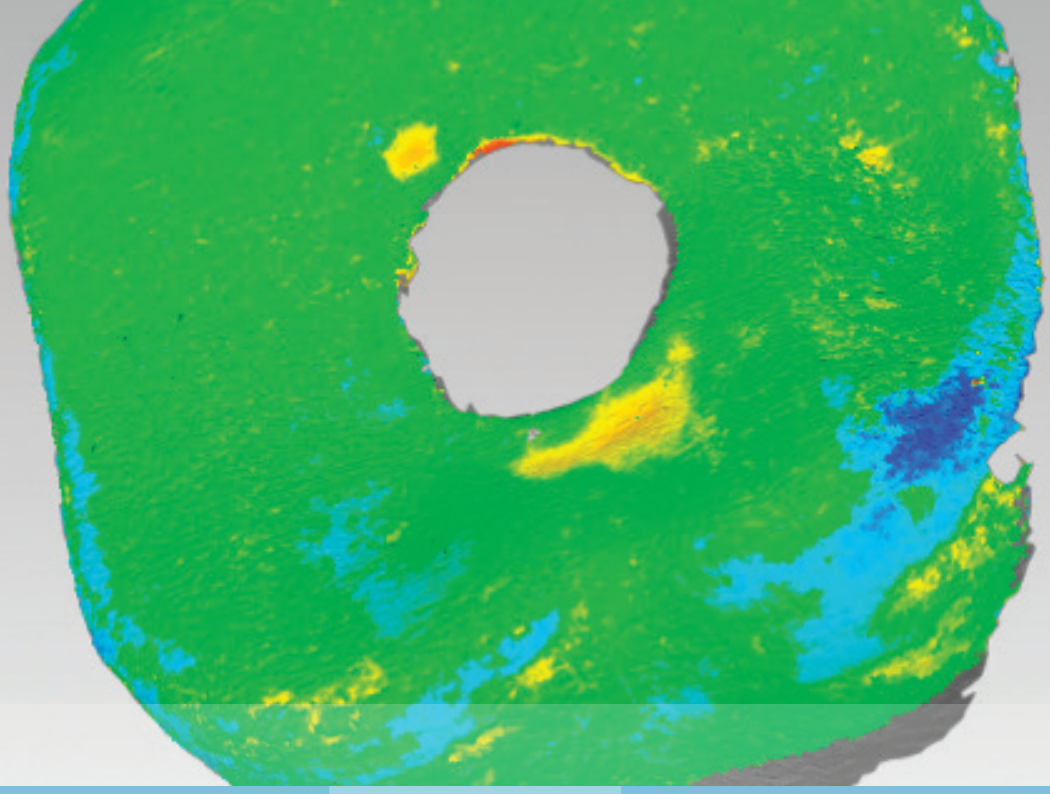

5

Wear measurement of dental tissues and materials in clinical studies: A systematic review

Claudine P. Wulfman [1]
Vinciane Koenig [2]
Amélie K. Mainjot [2]

[1] *Unité de Recherches en Biomatériaux Innovants et Interfaces (URB2i) – EA4462, Faculté de Chirurgie Dentaire, Université Paris Descartes, Sorbonne Paris Cité and Service d'odontologie, Hôpital Albert Chenevier, Assistance Publique – Hôpitaux de Paris, France.*

[2] *Dental Biomaterials Research Unit (d-BRU) and Department of Fixed Prosthodontics, Institute of Dentistry, University of Liège (ULiège) and University of Liège Hospital (CHU), Liège, Belgium.*

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ABSTRACT

Objectives: This study aims to systematically review the different methods used for wear measurement of dental tissues and materials in clinical studies, their relevance and reliability in terms of accuracy and precision, and the performance of the different steps of the workflow taken independently.
Methods: An exhaustive search of clinical studies related to wear of dental tissues and materials reporting a quantitative measurement method was conducted. MedLine, Embase, Scopus, Cochrane Library and Web of Science databases were used. Prospective studies, pilot studies and case series (>10 patients), as long as they contained a description of wear measurement methodology. Only studies published after 1995 were considered.
Results: After duplicates' removal, 495 studies were identified, and 41 remained for quantitative analysis. Thirty-four described wear-measurement protocols, using digital profilometry and superimposition, whereas 7 used alternative protocols. A specific form was designed to analyze the risk of bias. The methods were described in terms of material analyzed; study design; device used for surface acquisition; matching software details and settings; type of analysis (vertical height-loss measurement vs volume loss measurement); type of area investigated (entire occlusal area or selective areas); and results.
Significance: There is a need of standardization of clinical wear measurement. Current methods exhibit accuracy, which is not sufficient to monitor wear of restorative materials and tooth tissues. Their performance could be improved, notably limiting the use of replicas, using standardized calibration procedures and positive controls, optimizing the settings of scanners and matching softwares, and taking into account unusable data.

Keywords: *in vivo*, profilometry, measurement method, restorative materials, prosthodontics, tooth tissues, accuracy, precision, scanners, matching softwares

1. INTRODUCTION

Wear is a major issue for dental tissues that are submitted to abrasion, attrition, and erosion processes [1–4] and for dental materials that need to be sufficiently wear resistant without being prejudicial for the opposing teeth [5,6]. *In vitro* studies are interesting screening tools; they are easily performed and cheaper and faster than clinical studies, but they encounter numerous difficulties in reproducing the complexity of the oral environment. Moreover, there is no standardization regarding *in vitro* test conditions, and the prediction of clinical wear with *in vitro* studies is still debatable [7–9].

On the other hand, the literature reports different methods to evaluate dental tissue and material wear clinically. Many authors scaled wear intraorally by qualitative visual assessment, using different indexes [10,11], while some others evaluated it indirectly on casts, comparing them with a panel of standard models as a wear scale. Those methods can be considered as semi-quantitative methods [12–14]; they do not allow for an accurate wear quantification and tend to underestimate the amount of substance loss [15]; therefore, several indirect methods, which measure wear *in vitro* on tooth replicas, were introduced. The first indirect methods, developed in the 1970s, used stereomicroscopes [16] or microscopes coupled with contact gauges to measure the evolution of vertical substance loss at some defined points in time [17]. Those methods encountered limitations related to the reproducibility of measurement point localization and sample positioning at successive evaluation times, since even a slight angulation of the specimen in its holder can engender measurement imprecision. Therefore, in current techniques, which were introduced in the 1980s and 1990s, the entire occlusal surface is evaluated by digital profilometry. The sample is placed on a computer-controlled stage, and the full occlusal anatomy is scanned either with a contact gauge, or a non-contact electrical gauge [18], or by optical reflection (optical digital profilometry [ODP]), giving a cloud of measurement points with proper x, y, and z coordinates [19,20]. The quality of the scanning

procedure is influenced by the following parameters: the width of the laser spot/stylus size, the resolution in the three axes x/y/z, the scanning step, the angulation of the sample, the depth range, and the optical properties of the material. The obtained cloud of points is subsequently treated with mathematical models to extrapolate the whole surface. The digital reconstructions obtained at each evaluation time are then superimposed with metrology software, called matching software (MS), which allows for wear quantification, statistical analysis, and production of scaled, colored images. The superimposition concept consists of achieving the best fit possible between two reconstructions, performing numerous iterations of combined rotations and translations in areas that suffered the minimum of wear. The fit is obtained by minimizing the root mean square (RMS) differences in the z-axis on a minimum of points.

Currently, different protocols have been proposed in the literature, with significant variations in terms of replica-manufacturing procedure, scanner, or MS characteristics and settings. However, those parameters can significantly influence wear measurement accuracy and precision. Accuracy characterizes the ability of a measurement device to deliver a value as close as possible to the true value; it is often described as the mean of multiple measures, with standard deviation standing for precision, which characterizes the repeatability of the measurement system [21].

Consequently, the objectives of this work are to systematically review the specific characteristics of the different methods used for clinical wear measurement of dental tissues and materials, their relevance and reliability in terms of accuracy and precision, and the performance of the different steps of the workflow taken independently.

2. METHODS

Search strategy and data extraction followed the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) statement [22]. The review was registered in PROSPERO under registration number CRD42016042103. Assessment of risk of bias was focused on issues related to methodology.

2.1. Eligibility criteria and search strategy

The search strategy (Table 5.1) was conducted to include prospective studies on restorative materials as natural teeth, reporting a method of quantitative measurement of wear. Pilot studies and case series (>10 patients), as long as they report a detailed description of wear measurement methodology, were included. However, publications before 1995, literature reviews, abstracts, publications in a language different from English or French, publications relative to qualitative assessment of wear, and *in vitro* studies were excluded. Five databases were screened: MedLine (PubMed), Embase, Scopus, the Cochrane Library, and the Web of Science. The references cited in the articles included were also checked.

2.2. Study selection

Two reviewers (CW and VK) carried out the literature search independently until November 2017. All titles and abstracts of articles found were analyzed and selected in accordance with the eligibility criteria. Those that appeared to meet the inclusion criteria, or in which there was insufficient data in the title and the abstract to make a decision, were selected for full analysis. The two reviewers assessed the full-text articles independently. Any disagreement on the eligibility of studies included was resolved through discussion and consensus.

PubMed (11/30/2017)

#1 All-ceramic* OR Artificial Teeth OR Ceramic OR Ceramic Crown OR Ceramic Crowns OR Composite OR Composites OR Composite Restoration OR Composite Restorations OR Crown OR Crowns OR Dental Compo site* OR Dentin OR Dental Material OR Dental Materials OR Dental Resin Composite OR Denture Teeth OR Denture Tooth OR Esthetic Restoration OR Glass Ceramic OR Glass Ionomer OR Lithium Di silicates glass ceramic OR Metal Ceramic OR Polymer OR Polymers OR Dental Prosthesis OR Prosthodontic OR Resin OR Resin Composite OR Dental Restoration OR Worn Dentition OR Tooth OR Y-TZP OR Zirconia OR Zirconium OR Zirconium Oxide OR Dental Enamel[MeSH Terms] OR Ceramics*[MeSH Terms] OR Compomer*[MeSH Terms] OR Composite Resin*[MeSH Terms] OR Crown*[MeSH Terms] OR Dental Porcelain*[MeSH Terms] OR Dental Prosthesis*[MeSH Terms] OR Dental Veneer*[MeSH Terms] OR Denture, Complete*[MeSH Terms] OR Denture, Partial, Fixed*[MeSH Terms] OR Denture, Partial, Removable*[MeSH Terms] OR Glass ionomer Cement* [MeSH Terms] OR Inlay*[MeSH Terms] OR Metal Ceramic Alloy*[MeSH Terms] OR Tooth, Artificial*[MeSH Terms] OR Zirconium*[MeSH Terms] NOT Hip NOT Arthroplast*[MeSH Terms] NOT Breast*[MeSH Terms] NOT Lens

#1 AND #2 AND #3 AND #4

Embase

#1 'allceram' OR 'tooth' OR 'dentin' OR 'biomedical and dental material' OR 'ceramics'/de OR 'complete denture'/de OR 'compomer'/de OR 'composite material'/de OR 'dental material'/de OR 'dental porcelain' OR 'dental veneer'/ de OR 'denture'/de OR 'enamel'/de OR 'glass ceramics' OR 'glass ionomer'/de OR 'lithia disilicate' OR 'partial denture'/de OR 'resin'/de OR 'resin cement'/ de OR 'resin composite' OR 'tooth crown'/de OR 'tooth prosthesis'/de OR 'yttria stabilized tetragonale zirconia' OR 'zirconium'/de OR 'zirconium oxide'/ de NOT 'total hip prosthesis'/de NOT 'orthopedic implant'/de NOT 'total knee replacement'/de NOT 'hip':ab,ti

Table 5.1 *Electronic databases and search strategy.*

#2 3D Measurement* OR 3D Scanner* OR 3D Superimposition* OR Attrition* OR Erosion* OR Laser Scan* OR Material* Testing OR Measurement* OR Metrolog* OR Profilomet* OR Quantitative Measurement* OR Surface Mapping OR Surface Matching OR Image Processing, Computer-Assisted/instrumentation [MeSH Terms] OR Laser/statistics and numerical data[MeSH Terms] OR Materials Testing/methods* [MeSH Terms] OR Replica Techniques[MeSH Terms] OR Photogrammetry/statistics and numerical data [MeSH Terms]	#3 Clinical Wear OR Dental Restoration* Wear OR Dental Wear OR Dental Wear Measurement* OR Dental Wear Quantification* OR Enamel Wear OR *In Vivo* Wear OR Occlusal Wear OR Restoration* Occlusal Wear OR Tooth Wear OR Toothwear OR Tribology OR Wear OR Wear Measurement* OR Wear Stud* OR Wear Testing OR Wear Volume OR Dental Restoration Wear[MeSH Terms] OR Tooth Abrasion*[MeSH Terms] OR Tooth Attrition*[MeSH Terms] OR Tooth Erosion*[MeSH Terms] OR Tooth Wear[MeSH Terms] OR Tooth Wear/statistics and numerical data[MeSH Terms] OR Tooth Wear/diagnosis[MeSH Terms] NOT Tooth Wear/rehabilitation[MeSH Terms] NOT Tooth Wear/therapy[MeSH Terms]	#4 Clinical study OR *In vivo* Study OR Prospective study OR Clinical Trial[MeSH Terms] OR Cohort Stud*[MeSH Terms] OR Controlled Clinical Trial[MeSH Terms] OR Follow-up Stud*[MeSH Terms] OR Longitudinal Stud*[MeSH Terms] OR Multicenter Stud* [MeSH Terms] OR Pilot Stud*[MeSH Terms] OR Prospective Stud*[MeSH Terms] OR Randomized Controlled Trial[MeSH Terms]

#2 '3d measurement*':ab,ti OR '3d scanner*':ab,ti OR '3d superimposition*':ab,ti OR 'attrition*':ab,ti OR 'erosion*':ab,ti OR 'laser scan*':ab,ti OR 'measurement*':ab,ti OR 'metrolog*':ab,ti OR 'profilomet*':ab,ti OR 'surface mapping':ab,ti OR 'surface matching':ab,ti OR 'tribology':ab,ti OR 'wear volume':ab,ti OR 'accuracy'/de OR 'algorithm'/de OR 'calibration'/de OR 'computer program'/de OR 'computer system'/de OR 'image processing'/de OR 'image subtraction'/de OR 'laser'/de OR 'materials testing'/de OR 'odontometry'/de OR 'quality control'/de OR 'quantitative analysis'/de OR 'signal processing'/de OR 'three dimensional imaging'/de OR 'photogrammetry'/de	#3 'wear':ab,ti OR 'dental restoration wear'/de OR 'restoration occlusal wear':ab,ti	#4 'clinical research'/de OR 'clinical trial'/de OR 'cohort analysis'/de OR 'controlled clinical trial'/de OR 'follow-up'/de OR '*in vivo* study'/de OR 'longitudinal study'/de OR 'multicenter study'/de OR 'pilot study'/de OR 'prospective study'/de OR 'randomized controlled trial'/de NOT '*in vitro*'

Scopus

#1 TITLE-ABS-KEY(all ceramic) OR TITLE-ABS-KEY(tooth) OR TITLE-ABS-KEY(dentin) OR TITLE-ABS-KEY(composite restoration) OR TITLE-ABS-KEY(dental material) OR TITLE-ABS-KEY(worn dentition) OR TITLE-ABS-KEY(zirconia) OR INDEXTERMS(dental enamel) OR INDEXTERMS(ceramics) OR INDEXTERMS(compomers) OR INDEXTERMS(composite resins) OR INDEXTERMS(crowns) OR INDEXTERMS(dental porcelain) OR INDEXTERMS(dental prosthesis) OR INDEXTERMS(dental prosthesis) OR INDEXTERMS(dental veneers) OR INDEXTERMS(denture, complete) OR INDEXTERMS(denture, partial, fixed) OR INDEXTERMS(denture, partial, removable) OR INDEXTERMS(glass ionomer cements) OR INDEXTERMS(metal ceramic alloy) OR INDEXTERMS(tooth, artificial) OR INDEXTERMS(zirconium) AND NOT TITLE-ABS-KEY(hip) AND NOT TITLE-ABS-KEY(knee)

#1 AND #2 AND #3 AND #4 AND PUBYEAR AFT 1994

Cochrane Library

#1 tooth:ti,ab,kw
#2 dentin:ti,ab,kw
#3 all ceramic:ti,ab,kw
#4 composite restoration:ti,ab,kw
#5 dental material:ti,ab,kw
#6 worn dentition:ti,ab,kw
#7 zirconia:ti,ab,kw
#8 MeSH descriptor: [Dental Enamel] explode all trees
#9 MeSH descriptor: [Ceramics] explode all trees
#10 MeSH descriptor: [Compomers] explode all trees
#11 MeSH descriptor: [Composite Resins] explode all trees
#12 MeSH descriptor: [Crowns] explode all trees
#13 MeSH descriptor: [Dental Porcelain] explode all trees
#14 MeSH descriptor: [Dental Prosthesis] explode all trees
#15 MeSH descriptor: [Dental Veneers] explode all trees
#16 MeSH descriptor: [Denture, Complete] explode all trees
#17 MeSH descriptor: [Denture, Partial, Fixed] explode all trees
#18 MeSH descriptor: [Denture, Partial, Removable] explode all trees
#19 MeSH descriptor: [Glass Ionomer Cements] explode all trees
#20 MeSH descriptor: [Inlays] explode all trees
#21 MeSH descriptor: [Metal Ceramic Alloys] explode all trees
#22 MeSH descriptor: [Tooth, Artificial] explode all trees
#23 MeSH descriptor: [Zirconium] explode all trees
#24 #1 or #2 or #3 or #4 or #5 or #6 or #7 or #8 or #9 or #10 or #11 or #12 or #13 or #14 or #15 or #16 or #17 or #18 or #19 or #20 or #21 or #22 or #23
#25 3D measurement:ti,ab,kw
#26 3D scanner:ti,ab,kw
#27 3D superimposition:ti,ab,kw
#28 profilometry:ti,ab,kw

Web of Science

TOPIC: (dental material* OR enamel) AND **TOPIC:** (wear*) AND **TOPIC:** (measurement*) AND **TOPIC:** (clinical)
Analysis: **PUBLICATION YEARS**: (2016 OR 2015 OR 2014 OR 2013 OR 2012 OR 2011 OR 2010 OR 2009 OR 2008 OR 2007 OR 2006 OR 2005 OR 2004 OR 2003 OR 2002 OR 2001 OR 2000 OR 1999 OR 1998 OR 1997 OR 1996 OR 1995)

#2 TITLE-ABS-KEY(3D measurement) OR TITLE-ABS-KEY(3D scanner) OR TITLE-ABS-KEY(3D superimposition) OR TITLE-ABS-KEY(profilometry) OR TITLE-ABS-KEY(surface matching) OR TITLE-ABS-KEY(quantitative measurement) OR INDEXTERMS(image processing, computer assisted) OR INDEXTERMS (lasers) OR INDEXTERMS(materials testing) OR INDEXTERMS(replica techniques) OR INDEXTERMS(tooth abrasion) OR INDEXTERMS(tooth attrition) OR INDEXTERMS(tooth erosion) OR INDEXTERMS(photogrammetry)	#3 TITLE-ABS-KEY (wear) OR INDEXTERMS (dental restoration wear) OR INDEXTERMS (tooth wear)	#4 INDEXTERMS(clinical trial) OR INDEXTERMS(cohort studies) OR INDEXTERMS(controlled clinical trial) OR INDEXTERMS(longitudinal studies) OR INDEXTERMS(multicenter studies) OR INDEXTERMS(pilot projects) OR INDEXTERMS(prospective studies) OR INDEXTERMS(randomized controlled trial) AND NOT INDEXTERMS(*in vitro*) AND NOT INDEXTERMS(laboratory studies) AND NOT TITLE-ABS-KEY (*in vitro*) AND NOT TITLE-ABS-KEY (laboratory studies) AND NOT TITLE-ABS-KEY(*in situ*)	

#29	surface matching:ti,ab,kw	#44	MeSH descriptor: [Clinical Trial] explode all trees
#30	surface mapping:ti,ab,kw	#45	MeSH descriptor: [Cohort Studies] explode all trees
#31	quantitative measurement:ti,ab,kw	#46	MeSH descriptor: [Controlled Clinical Trial] explode all trees
#32	MeSH descriptor: [Photogrammetry] explode all trees	#47	MeSH descriptor: [Longitudinal Studies] explode all trees
#33	MeSH descriptor: [Image Processing, Computer-Assisted] explode all trees	#48	MeSH descriptor: [Multicenter Study] explode all trees
#34	MeSH descriptor: [Lasers] explode all trees	#49	MeSH descriptor: [Pilot Projects] explode all trees
#35	MeSH descriptor: [Materials Testing] explode all trees	#50	MeSH descriptor: [Prospective Studies] explode all trees
#36	MeSH descriptor: [Replica Techniques] explode all trees	#51	MeSH descriptor: [Randomized Controlled Trial] explode all trees
#37	#25 or #26 or #27 or #28 or #29 or #30 or #31 or #32 or #33 or #34 or #35 or #36	#52	*in vivo*:ti,ab,kw
#38	MeSH descriptor: [Dental Restoration Wear] explode all trees	#53	#44 or #45 or #46 or #47 or #48 or #49 or #50 or #51 or #52
#39	MeSH descriptor: [Tooth Abrasion] explode all trees	#54	#24 and #37 and #43 and #53
#40	MeSH descriptor: [Tooth Erosion] explode all trees		
#41	MeSH descriptor: [Tooth Attrition] explode all trees		
#42	MeSH descriptor: [Tooth Wear] explode all trees		
#43	#38 or #39 or #40 or #41 or #42		

Table 5.1 *(Continued)*

2.3. Data extraction

A data collection form was pilot-tested using a sample of 10 studies to ensure that the criteria fit the research question. One review author (CW) extracted the data, and the second (VK) checked it.

Reviewers tabulated the following data from the included articles: (1) material or dental tissue analyzed; (2) study details, including year of publication and author(s); (3) study method, including design and number of patients; (4) details about the device used for surface acquisition; (5) matching software (MS) details and setting procedure; (6) type of analysis (2D, meaning vertical height loss measurement, or 3D, meaning volume loss measurement); (7) type of area investigated (entire occlusal area or selective measurement areas); (8) quantitative results.

When a research team reported intermediary results of an ongoing study, data from all reports were compiled.

Because of the high degree of heterogeneity in terms of materials studied and methodologies, it was considered inappropriate to conduct a meta-analysis.

2.4. Assessment of risk of bias

A specific form was developed to report risk of bias with a focus on wear measurement methodology. The following parameters were registered: (1) existence of a measurement chain calibration, allowing accurate measurement of the workflow used, in the study itself or in a previous publication from the same research team, using the same protocol; (2) description of matching software (MS) settings; (3) use of enamel as a control of the measurement chain; (4) reporting of unusable data, for example, due to problems encountered with the impression, cast, or scan quality. If one

of these items was reported in the article, the corresponding entry was recorded as "Y" (yes). If the information was not found, the entry was recorded as "N" (no). Articles reporting only one or two items encoded as yes were classified as having a high risk of bias, three items as a medium risk of bias and four items as a low risk of bias.

3. RESULTS

3.1. Study selection

After database screening and removal of duplicates, 495 studies were identified (Fig. 5.1). After title screening, 250 studies remained, and this number was reduced to 63 after careful examination of the abstracts. The full texts of those 63 studies were assessed to check whether they were eligible. Among them, 12 were excluded due to the following reasons: (1) the study was not prospective [7,23]; (2) the quantification aimed at the measurement of dental displacement after orthodontic treatment [24,25]; (3) the study focused on wear analysis of restoration margins [26,27]; (4) the measurement was semi-quantitative, using comparison with a panel of standard models [28–33]. After merging of studies related to successive reports at different time periods, 41 studies were identified.

3.2. Study characteristics

In 34 studies, quantitative wear measurement was performed on the entire occlusal surface, using digital profilometry (DP), most often with optical scanners (ODP), followed by superimposition of the scans at the different observation times with an MS. In the seven other studies, the quantitative wear measurement was performed only on some selected occlusal reference

points using metallic disc indexes [34–36], contact gauge [37], crown height measurement with an optical microscope [38,39], or image analysis software [40]. All studies are detailed and listed according to the material depicted in Tables 5.2 and 5.3. A large variation in protocols and devices used was observed between the different studies. The studied materials included ceramics

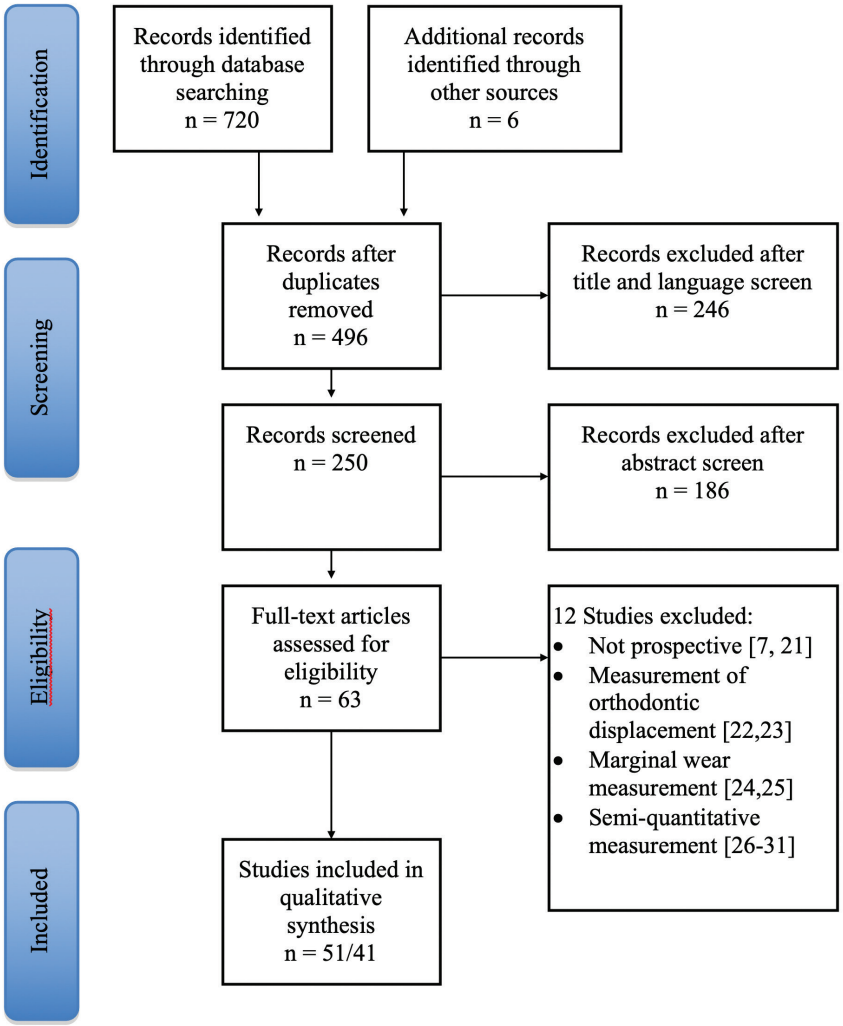

Fig 5.1 *Flow diagram of study identification.*

(*n* = 10 studies), composites (*n* = 16 studies), denture tooth resin (*n* = 7 studies), and enamel (*n* = 8 studies). Three studies compared composites to ceramics.

In most studies, wear measurement was related to the restorative materials (*n* = 25 studies), while 2 studies focused on antagonistic teeth and six studies on both restorative materials and antagonistic teeth. Regarding the type of analysis, 22 studies reported 2D analysis (vertical height loss); four studies reported 3D (volumetric loss) and 12 studies reported both analyses. Moreover, two studies presented their results according to tooth height and another one according to surface measurement. It must be noted that 2D analysis can be realized with all MS, while 3D is only possible with some specific softwares, for example, Match 3D software (Willytec, Feldkirchen-Westerham, Germany) allows for 3D measurement; Geomagic Control (Geomagic, Inc., Morrisville, NC, USA) does not. Finally, two studies showed a strong correlation between 2D and 3D measurements [41,42], and some authors reported that both are useful [21,43]. Partisans of 2D measurement reported that this analysis is independent of the dimension of the tooth, while volumetric loss is influenced by tooth size [5].

Regarding the type of area analyzed, two approaches were used [44]: the measurement of the entire occlusal area or selective measurement areas, some authors measuring wear on occlusal contact areas (OCA), and others on contact-free occlusal areas (CFOA) [45,46]. Furthermore Chadwick et al. proposed to measure the tooth surface percentage that is submitted to wear, introducing a 5-point scale for epidemiological studies [47].

3.3. Assessment of risk of bias

The risk of bias for each study respectively is reported in Table 5.4. Thirteen studies of 41 exhibited a low risk of bias,16 a medium risk, and 12 a high risk.

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Ceramic	Anselm Wiskott et al., University of Geneva [70]	RCT	12 (10)	Au Alloy + Feldspathic (SuperPorcelain EX-3, Noritake)/ Composite (Colombus, Cendres et Métaux)/Resin (SR Ivocron, Ivoclar Vivadent)	Home-made mechanical
Ceramic	Esquivel-Upshaw et al., University of Florida [6]	Prospective	30 (21)	e.max Press (Ivoclar Vivadent). Measurements on opposing teeth	LaserScan 3D (Willytec) Optical
Ceramic	Esquivel-Upshaw et al., University of Florida College of Dentistry Silva et al., New York University College of Dentistry [85–87]	RCT	36 (31)	Pd alloy + IPS d.SIGN veneer (Ivoclar Vivadent), IPS e.max Press core (Ivoclar Vivadent), IPS Empress 2 core and glass-ceramic veneer IPS Eris (Ivoclar Vivadent) + antagonist, control	etkon es1 (formerly Willytec GmbH, now Straumann CADCAM) Optical

Table 5.2 *Studies using profilometry and superimposition software.*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
MATLAB	Vertical loss (mean) volumetric loss	2	5 µm after calibration by scan superim-position but vertical resolution not mentioned.	Ceramic and composite: 12–14 µm y^{-1}, volume 10–12 μm^3 μm^2 y^{-1}, resin: 50 µm y^{-1}, volume 45 μm^3 μm^2 y^{-1}
Match 3D (Willytec)	Vertical loss (mean)	1	Not provided	Premolars: 88.4 µm Molars: 88.3 µm
Match 3D	Vertical loss (mean) volumetric loss	3	Not provided	Ceramics: 1.48 (0.2) mm^3, 1.31 (0.17) mm^3, 1.06 (0.12) mm^3 Antagonist enamel: 1.1 (0.1) mm^3, 1.02 (0.2) mm^3, 0.8(0.09) mm^3

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Ceramic	Etman et al., University of Saskatchewan, Saskatoon, Canada, King's College London Dental Institute [54]	RCT	90 (48)	Gold alloy + IPS (Ivoclar-Vivadent), ProceraAllCeram (Nobel Biocare), Experimental lithium disilicate + antagonists	Keyence LC-2400 series laser displacement meter Optical
Ceramic	Krämer et al., University of Dresden, University of Munich, University of Erlanger-Nurember [56]	Prospective	17 (10)	IPS Empress (Ivoclar Vivadent) + Enamel	LaserScan 3D Optical
Ceramic	Lohbauer et al., University of Erlanger and University of Aachen, Germany [43]	Prospective	14 (14)	Zirconia (Lava Plus, 3M ESPE) + antagonists	CT 100 (Cybertechnologies, Ingolstadt) Optical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Scan-Surf	Vertical loss (mean, maximum, minimum) OCA	2	Not provided	Restorations 6 months: 87.06 (2.96)–143.6 (9.47) µm, 1 year: 116.3 (4.74)–201.18 (10.22) µm, 2 years: 176 (3.93)–321.6 (12,79) µm, Wear metal–ceramic < experimental < Procera
Match 3D	Vertical loss restoration surface for ceramic OCA for enamel	8	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	Ceramic 4 years: 78 µm, 8 years: 116 µm, enamel 4 years: 120 µm, 8 years: 238 µm
Scan CT V 8.4, Cyber-technologies	Vertical loss (maximum) volumetric loss (mean) OCA	2	Not provided	Enamel antagonist contacts: 0.361 mm^3 and mean maximum vertical loss 0.204 mm Ceramic contacts: 0.333 mm^3 and 0.145 mm

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Ceramic	Mundhe et al., All India Iae of Medical Sciences [57]	Split-mouth	20 (10)	Enamel antagonist to : zirconia (LAVA, 3M ESPE), Nickel alloy + feldspathic, enamel control	Smart SCAN3D (HE scanner; Breukmann) Optical
Ceramic	Sorensen et al., University of Portland [88]	Prospective	60 (57)	Lithium disilicate ceramic, Empress 2 (Ivoclar Vivadent) + antagonists	Tooth profiling system (MTS) Mechanical
Ceramic	Stober et al., University Hospital Heidelberg [62,89]	Split-mouth	20 (20)	Zirconia (Zenostar Zr Translucent), antagonist enamel, control	LaserScan 3D Optical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
3D software (Polyworks)	Vertical loss	1	No calibration. 9 µm-vertical resolution	Feldspathic: 69.20 (4.10) µm for premolar and 179.70 (8.09) µm for molar, zirconia: 42.10 (4.30) µm for premolar and 127.00 (5.03) µm for molar, enamel: 17.30 (1.88) µm for premolar and 35.10 (2.60) µm for molar
Ansur Software	Volumetric loss (mean)	6–18 months	Not provided	Opposite teeth : 0.0701 (0.121) mm^3 Ceramic: 0.0268 (0.370) mm^3
Match 3D	6 months : mean vertical loss, 2 years : mean / max vertical loss, OCA	6 months, 2 years	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	6 months: zirconia: 10 (5) µm, antagonist enamel: 33 (32) µm, control: 10 (6) µm, 2 years: zirconia: 14/60 µm antagonist enamel: 46/151 µm control: 19–26/75–115

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Ceramic	Supputa-mongkol et al. [90], University of Bangkok, University of Florida	Prospective	30 (30)	lithium disilicate-based all-ceramic system (Ivoclar Vivadent) + antagonists	etkon es1
Composite crowns	Ohlman et al. J Dent, Heidelberg University Hospital [69,91]	RCT	120 (66)	Experimental polymeric material (Ivoclar Vivadent) with or without Vectris (Ivoclar Vivadent), high-gold alloy + IPS d.Sign (Ivoclar Vivadent)	LaserScan 3D Optical
Composite crowns	Stober et al., University of Heidelberg [92]	Prospective	114 (74)	Artglass (Heraeus Kulzer	LaserScan 3D Optical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Match 3D	Volumetric loss (mean)	1	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	Ceramic: 0.19 (0.065) mm^3 for premolar and 0.34 (0.08) mm^3 for molar sites Opposing enamel: 0.21 (0.06) mm^3 for premolar and 0.50 (0.22) mm^3 for molar
Match 3D	Vertical loss (mean) Occlusal surface + OCA	1 and 2 years	A 10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	1 year: occlusal surface: 8.1 (9.7) µm, 7.0 (9.1) µm, 3.4 (6.3) µm OCA: 44.7 (37.6) µm, 39.2 (34.1) µm, 26.8 (28.6) µm, 2 years: occlusal surface: 19 (18.5) µm, 24.3 (31.5) µm, 7 (8.8) µm OCA: 81.8 (67.1) µm, 76.8 (49.2) µm, 38.5 (31) µm
Match 3D	Vertical loss (mean) OCA	1 and 2 years	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	1 year: occlusal surface: PM 48 (79) M 72 (119) µm OCA: PM 54 (62) M 105 (140) µm 2 years: occlusal surface: PM 62 (47) M 97 (89) µm OCA: PM 74 (56) M114 (102) µm

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Composite crowns	Zenthöfer al., University of Heidelberg [93]	RCT	120 (66)	Trend HP (Vivadent), Vectris (Vivadent), IPS d.Sign (Ceramic) (Vivadent)	LaserScan 3D Optical
Composite	Cetin et al., Selcuk University, Konya, Turkey [49]	RCT	100 (54)	Filtek Supreme XT (3M ESPE), Tetric EvoCeram (Ivoclar Vivadent), Aelite Aesthetic (Bisco), Estenia (Kuraray), Tescera (Bisco)	3D Optical Scan System (Breuck-mann)
Composite	DeLong et al., University ofMinnesota School of Dentistry [45]	Prospective	12 (10)	SureFil (Caulk, Dentsply)	University of Minnesota contact profiling system (UMN) Mechanical
Composite	Krämer et al., University Medical Center Giessen and Marburg [52]	Prospective	68 (30)	Grandio (Voco), Tetric Ceram (Ivoclar Vivadent	Rapid (Thome Präzision) Mechanical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Match 3D	Vertical loss (mean) Occlusal surface + OCA	3	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	Trend: 41.8 (55.3) µm OCA: 131.4 (102.81) µm Vectris: 39.6 (39.5) µm OCA: 104.1 (61.9) µm Ceramic: 24.4 (36.5 µm OCA: 85.8 (74.3) µm
RapidForm	Vertical loss (maximum, minimum, mean), volumetric loss OCA	1	Not provided	6 months (mean vol loss): 0.058 (0.01)–0.088 (0.02) mm^3, 1 year: 0.09 (0.02)–0.132 (0.03) mm^3
Ansur Software	Vertical loss (mean) Volumetric loss (mean) OCA, CFOA	5	Not provided	All restorations: 27 (24) m, 0.532 (0.372) mm^3, OCA: 55 (20) µm, 0.373 (0.188) mm^3, CFOA: 18 (33) µm, 0.432 (0.499) mm^3
GOM Inspect	Vertical loss Restoration and restora-tion OCA	8	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	2 ans: 41 (30) µm (Grandio) and 44 (34) µm (tetric) on restorations, 54 (56) and 55 (44) µm (OCA) 4 ans: 66 (55) and 61 (38) µm, 100 (90) and 74 (56) µm (OCA) 8 ans: 108 (88) and 98 (53) µm, 135 (104) and 110 (58) (OCA)

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Composite	Lawson et al., University of Alabama at Birmingham [79]	RCT	120 (60)	Filtek Supreme Ultra Flowable Restorative (3M Espe), Filtek Supreme Ultra Universal Restorative (3M Espe)	Proscan 2000 (Scantron Industrial Products Ltd) Optical
Composite	Palaniappan et al., Catholic University of Leuven [41,46,94–96]	RCT	35 (16) 32 (49) 86 (31)	Tetric-C, Tetric-EC (Ivoclar), Gradia-DP (GC), Filtek Supreme (3M Espe), Z100 (3M Espe)	LaserScan 3D Optical
Composite	Perry et al. [15], Tufts University College of Dentistry	Prospective	21 patients	Pertac (ESPE) and TPH (Dentsply)	LaserScan 3D Optical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Proform	Volumetric loss (restoration)	2	Not provided	3.16 (2.38) mm^3 for the flowable composite 3.43 (2.50) mm^3 for the conventional composite
Match 3D	OCA Vertical loss (mean) volumetric loss (total, enamel, restorative) CFOA* Vertical loss (mean)	5	Not provided	OCA 6 months: 32 (9)–49 (16) µm, 0.073 (0.05)-0.664 (0.4) mm^3, 1 year: 43 (17)–72 (28) µm, 0.157 (0.1)–0.849 (0.3) mm^3, 2 years: 54 (20)–98 (38) µm, 0.304 (0.1)–1.207 (0.6) mm^3, 3 years: 64 (75)–125 (45) µm, 0.415 (0.2)–1.542 (0.8) mm^3, 5 years: 77 (25)–139 (44) µm, 1.14 (0.7)–1.89 (1.0) mm^3, CFOA* Class I cavities, 6 months: 8 (12)–40 (16) µm, 1 year: 37 (17)–54 (25), 2 years: 40 (18)–67 (26), 3 years: 44 (19)–96 (28), 4 years: 50 (16)–116 (28), 5 years: 60 (18)–119 (31)
Match 3D	Vertical loss (mean) volumetric loss	2	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	83 µm

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Composite	Pesun et al., University of Minnesota [97]	Pilot-study	10 (10)	Z100 (3M)	UMN Mechanical
Composite	Peters et al., University of Michigan, University of Minnesota [65]	Results from RCT	14 patients	P-10 (3M), P30 et Miradapt (Johnson and Johnson)	UMN Mechanical
Composite	Rosin et al. [71,98], University of Greifswald and Eastman dental institute, University college of London	Prospective	33 restorations from 356 (117)	Definite (Degussa)	LaserScan 3D (Willytec) Optical
Composite	Söderholm et al., University of Florida, University of Leuven [66]	RCT	128 (25)	Eight experimental composites	LaserScan 3D Optical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Ansur Software	Vertical loss (maximum and mean) Volumetric loss Occlusal surface + OCA	1	7 µm after calibration by scan super-imposition, but without taking into account impression accuracy Vertical resolution not mentioned	Preparation maximum vertical loss: 204.8 (129.8) µm enamel OCA maximum vertical loss: 36.8 (10.1) µm
Ansur Software	Volumetric loss CFOA, Margins maximal vertical loss	5	7 µm after calibration by scan super-imposition, but without taking into account impression accuracy Vertical resolution not mentioned	1.6–3.1 mm^3
Match 3D	Vertical loss (mean)	6 months, 1 and 2 years	7.75 µm after calibration by scan super-imposition but vertical resolution not mentioned	6 months: PM 2.4 (13.5) µm M 11.7 (18.5) µm 1 year: PM 10 (11.6) µm M 22 (24.1) µm 2 years: PM 12.3 (11.3) µm M 21.4 (20.4) µm Wear rate: 8.5 (3.7) µm/month
Match 3D	Vertical loss (maximum and mean), volumetric loss	3	Not provided	

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Composite / Glass ionomer	Folwaczny et al., Ludwig-Maximilian University [50]	RCT	197 (37)	Tetric (Vivadent), Dyract (DeTrey Dentsply), Fuji II LC (GC Dental), Photac-Fil (Espe)	LaserScan 3D Optical
Denture teeth	Heintze et al., Ivoclar Vivadent, Medical University Innsbruck, University of Buffalo [64]	RCT	30 patients	DCL (Ivoclar Vivadent), Experimental material (Ivoclar Vivadent), NFC (Candulor)	etkon es1
Denture teeth	Heintze et al., Ivoclar Vivadent University of Buffalo and Heidelberg University Hospital [44]	RCT	89 patients	DCL (Ivoclar Vivadent), Experimental material (Ivoclar Vivadent)	etkon es1

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Match 3D	Vertical loss (mean)	3	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	Photac-Fil 44 (23) µm, Fuji II LC 45 (26) µm, Dyract 71 (47) µm, Tetric 18 (12) µm
Match 3D	Vertical loss (maximum) OCA	1	10 µm after calibration by scan super-imposition.	DCL: 225.9 (109.4) µm, EM: 211.2 µm (100.2), NFC: 140.4 (39.9) µm
Match 3D	Vertical loss (maximum and mean), OCA	2	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	>200 µm for both materials, more wear in the maxillary teeth than in the mandibular teeth, in the first molar teeth than in other posterior teeth, in the supporting cusps than in the nonsupporting cusps. The amount of wear did not depend on whether or not the lower dentures were supported by implants

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Denture teeth	Ohlman et al. Int J Prostho, Heidelberg University Hospital [69]	Prospective	30 patients	Vitapan (Vita Zahnfabrik)	LaserScan 3D Optical
Denture teeth	Schmid-Schwap et al. [42], Medical University of Vienna, University of Lausanne	Split-mouth, pilot study	28 patients	DCL (Ivoclar Vivadent), Experimental material (Ivoclar Vivadent)	LaserScan 3D Optical
Denture teeth	Stober et al. [68], Heidelberg University Hospital	Prospective	50 patients	Vitapan (VITA Zahnfabrik)	LaserScan 3D Optical
Denture teeth	Stober et al. Dent Mater, Heidelberg University Hospital [5]	RCT	12 patients	DCL (Ivoclar Vivadent), Experimental material (Ivoclar Vivadent)	Laserscan 3D, etkon es1

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
Match 3D	Vertical loss (mean)	6 months	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	Males 22.5 (15.8) µm, females (–18.7 (13.1)) µm, removable partial denture 22.8 (14.9) µm, complete denture 18.9 (13.2) µm Overdenture 17.4 (16.7) µm
Match 3D	Vertical loss (maximum) OCA Volumetric loss	1	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	First maxillary molar: DCL 0.212 µm, 2.05 mm^3, EM 0.221 m, 1.36 mm^3
Match 3D	Vertical loss (mean and maximum)	2	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	6 months: 8 (19), 92 (112), 1 year: 18 (34), 146 (148), 2 years: 40 (61), 226 (184)
Match 3D	Vertical loss (maximum) Occlusal surface + OCA	2	10 µm after calibration by scan super-imposition. 5 µm-vertical resolution	DCL: 142 (126) µm, OCA 187 (136) µm, experimental: 120 (81) µm, OCA 154 (82) µm

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Acquisition device
Enamel	Chadwick et al. [55], Dundee dental school and hospital, University of Newcastle	Prospective	250 patients	-	Home-made Electrical
Enamel	Pintado et al., University of Minnesota [51]	Prospective	18 patients	-	UMN Mechanical
Enamel	Rodriguez et al. [63], King's College London dental institute	Prospective	63 patients	-	Xyris 2000 TL (Taicaan) Optical
Enamel	Tantbijorn et al., University of Minnesota [99]	Parallel	18 patients	Patients presenting gastroeso-phageal reflux disease (GERD) and control	Lava Scan ST (ESPE) Optical

Table 5.2 *(Continued)*

Matching software (MS)	Type of analysis (investigated area)	Follow -up (years)	Workflow accuracy and precision	Quantitative results
SMADDA Home-made	Vertical loss used in a Tooth Surface Loss scale	1,5	4.4 µm after calibration by scan super-imposition. 1 µm vertical resolution	14% teeth showed wear, with 8% presenting a surface mostly unchanged but <15%, 2 teeth had 26–50% of the surface exhibiting loss, 1 teeth had more than 51% of the tooth surface exhibiting loss
Ansur Software	Vertical loss (mean) volumetric loss	2	7 µm after calibration by scan super-imposition, but without taking into account impression accuracy Vertical resolution not mentioned	1 year: 10.74 (24.25) µm 0.044 (0.251) mm^3, 2 years: 20.08 (29.59) µm 0.098 (0.331) mm^3
Geomagic qualify 11	Vertical loss (mean)	6 months, -1 year	15 µm after calibration by scan super-imposition but vertical resolution not mentioned	6 months: Tooth level 72% <15 µm, subject level 78% <15 µm, more wear on lower molars and upper incisors DCL: 142 (126) µm, OCA 187 (136) µm, experimental: 120 (81) µm, OCA 154 (82) µm
Cumulus Software (University of Minnesota)	Volumetric loss (mean)	6 months	7 µm after calibration by scan super-imposition, but without taking into account impression accuracy Vertical resolution not mentioned	GERD: 0.18 (0.12) mm^3 , Control: 0.06 (0.03) mm^3

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Method
Composite/ sealant	Bartlett et al., King's college London dental institute [34]	Split-mouth	17 patients	Helioseal Clear Chroma (Ivoclar- Vivadent)	Measurement of the vertical distance between the material and reference metallic discs
Composite/ adhesive	Sundaram et al., King's college London dental institute [35]	Split-mouth	19 patients	Seal & Protect (Dentsply)	Measurement of the vertical distance between the material and reference metallic discs
Denture teeth	Ogle et al., Lindquist and Davis, University of Buffalo [37,100]	RCT	67 patients	SR Ivoclar-Vivodent/ Orthotyp PE, Dentsply-Trublend SLM, Dentsply-Bioblend IPN	Measurement of vertical height of selected points
Enamel	Al-Omiri et al., University of Jordan and Queen's University Belfast [39]	Prospective	50 patients	-	Measure of teeth height from gingiva to incisal edge

Table 5.3 *Studies using alternative quantification methods.*

Devices	Type of analysis (investigated area)	Follow -up (years)	Quantitative results
Profilometer Keyence LC-2400 Optical	Vertical loss (mean) on three reference points UBSoft (UBM Messtechnik GmbH)	3, 6, 9, 12 and 20 months	6 months: sealant 50 (260) µm control 120 (114) µm 12 months: sealant 160 (350) µm control 110 (90) µm 20 months: sealant 290 (500) µm control 180 (270) µm
Profilometer Keyence LC-2400 Optical	Vertical loss (mean) on three reference points UBSoft (UBM Messtechnik GmbH)	3, 6, 9, 12 and 20 months	12 months: control 0.12 mm adhesive 0.10 mm 20 months: control 0.24 mm adhesive 0.21 mm
Computerized coordinate measurement system	Vertical loss on reference points, MXF 203 measurement device mecha-nical	6, 12, 24 and 36 months	3 years: Ivoclar/Orthotyp 229 µm Dentsply Trublend 242 µm Bioform 157 µm
CAD-CAM Laser Cercon System (Cercon Smart Ceramics, DeguDent Tool maker microscope (Stedall-Dowding Machine Tool Company)	Tooth height	1 year	Wear measurement after conventional impression: 130 (20) µm Wear measurement after digital impression: 68 (23) µm

Material or tissue analyzed	Study	Study design	Amount of restorations (patients)	Products	Method
Enamel	Al-Omiri et al., University of Jordan and Queen's University Belfast [38]	Prospective	20 patients	-	Measure of teeth height from gingiva to incisal edge
Enamel	Bartlett et al., Guy's Hospital, London and Birmingham University [36]	Parallel study	20 patients	-	Measurement of the vertical distance between the material and reference metallic discs
Enamel	Wood et al., University of Leeds [40]	RCT	31 patients	Cervical wear with and without occlusal adjustment	Measure of abfraction area on sliced casts

Table 5.3 *(Continued)*

Devices	Type of analysis (investigated area)	Follow -up (years)	Quantitative results
CAD-CAM Laser Cercon System (Cercon Smart Ceramics, DeguDent) Tool maker micros-cope (Stedall-Dowding Machine Tool Company)	Tooth height	6 months	Wear measurement after conventional impression: 582 (50) µm Wear measurement after digital impression: 165 (27) µm
3D topographical profilometer (Rank Taylor Hobson, Leicester, UK) Mechanical profilometer	Vertical loss on 20 reference points	6 months	Patients with erosion: 36.5 µm Controls: 3.7 µm
Stereomicro-scope (Type S Wild M3Z, Wild Heerburgg) Image analysis with Sigma Scan Software (SigmaScan Pro 5.0.0,Aspire Software International)	Progression of abfraction lesion area	6, 18 and 30 months	6 months: not adjusted 0.06 (0.069) mm^2 adjusted 0.08 (0.134) mm^2 18 months: not adjusted 0.142 (0.107) mm^2 adjusted 0.158 (0.162) mm^2 30 months: not adjusted 0.202 (0.140) mm^2 adjusted 0.225 (0.224) mm^2

Study	Calibration	Matching software setting details	Positive control	Limitations report	Risk of bias
Studies using profilometry and superimposition software					
Anselm Wiscott et al. [70]	Y	Y	N	Y	Medium
Cetin et al. [49]	N	N	N	Y	High
Chadwick et al. [55]	Y	Y	–	Y	Low
Delong et al. [45]	N	N	N	N	High
Esquivel-Upshaw et al. [6]	N	N	N	N	High
Esquivel-Upshaw et al. [85–87]	N	N	Y	Y	Medium
Etman et al. [54]	N	N	N	N	High
Folwaczny et al. [50]	Y	Y	N	Y	Medium
Heintze et al. [64]	Y	Y	-	N	Medium
Heintze et al. [44]	Y	Y	-	Y	Low
Krämer et al. [56]	Y	Y	N	Y	Medium
Krämer et al. [52]	N	N	N	N	High
Lawson et al. [79]	N	N	N	Y	High
Lohbauer et al. [43]	N	Y	N	Y	Medium
Mundhe et al. [57]	N	N	Y	N	High
Ohlman et al. [91,101]	Y	Y	N	Y	Medium
Ohlman et al. [69]	N	Y	-	Y	Medium
Palaniappan et al. [41,46,94–96]	N	Y	Y	Y	Medium
Perry et al. [15]	Y	Y	N	N	Medium
Pesun et al. [97]	Y	Y	Y	Y	Low
Peters et al. [65]	Y	Y	N	N	Medium
Pintado et al. [61]	Y	Y	-	Y	Low
Rodriguez et al. [63]	Y	Y	-	Y	Low
Rosin et al. [71,98]	Y	Y	N	N	Medium
Schmid-Schwap et al. [42]	Y	Y	-	Y	Low
Söderholm et al. [66]	N	Y	N	Y	Medium
Sorensen et al. [88]	N	N	N	N	High
Stober et al. [92]	Y	Y	N	N	Medium
Stober et al. [68]	Y	Y	-	Y	Low
Stober et al. [5]	Y	Y	-	Y	Low
Stober et al. [62,89]	Y	Y	Y	Y	Low
Supputamongkol et al. [90]	Y	Y	Y	Y	Medium
Tantbirojn et al. [99]	Y	N	Y	Y	Low
Zenthöfer et al. [93]	Y	Y	N	Y	Medium
Studies using alternative methods					
Al-Omiri et al. [38]	Y		-	N	High
Al-Omiri et al. [39]	Y		-	N	High
Bartlett et al. [36]	Y		Y	Y	Low
Bartlett et al. [34]	Y		Y	Y	Low
Ogle et al. [37,100]	Y		-	N	High
Sundaram et al. [35]	Y		Y	Y	Low
Wood et al. [40]	N		N	Y	High

Table 5.4 *Assessment of risk of bias in wear measurement analysis.*

3.4. Workflow calibration and accuracy measurement

Among the 41 included studies, 21 studies presented a detailed description of the calibration, accuracy, and precision of their measurement chain.

In most studies, measurements were performed on replicas issued from polyvinyl siloxane impressions cast in type IV stone. This combination was reported to provide a linear accuracy of 9 µm [48], depending on impression technique and tray choice. Other materials used included polyethers for impression [15, 49, 50] or epoxy resin for cast [51–53]. Etman et al. directly scanned impressions to avoid casting [54], as did Chadwick et al., who further coated impressions with a conductive paint to improve scanning [55].

Information about the different parameters influencing precision and accuracy of the scanning procedure (width of the laser spot/stylus size, resolution in the three axes x/y/z, scanning step, angulation of the sample, and depth range) was shown to be sparse. The x and y resolutions were usually included between 20 and 25 µm [6,19,56], combined with a scanning step in the same range (20-25 µm), whereas reported z resolution obtained with laser scans varied between 5 µm [15,56] and 9 µm [57] and was inferior to 1 µm with a mechanical profilometer [56].

Regarding accuracy of the workflow, some studies evaluated it by comparing iterated scans of an object or cast. The calibration procedure involves four steps: iterated superimposition of identical scans (superimposition process), superimposition of iterated scans of a standard model without displacing it from the positioning device (acquisition), superimposition of iterated scans of the standard model after removal and replacement in the positioning device (acquisition + superimposition), and, finally, superimposition of scans of duplicates of the standard model (complete procedure, including impression and casting) [58].

Superimposition of identical scans of a standard cast with Match 3D provided a precision of 0.5 µm in terms of measurement following the z-axis [58]. With Geomagic, acquisition and superimposition of a standard cast resulted in a 2.7 µm precision, whereas the same experiment resulted in a 14.8 µm precision with a cast obtained from successive impressions [59]. The same experiment performed with Match 3D resulted in 10 µm precision after successive impressions of dental tissues [19] and 7.75 µm for the gingiva, but using a different scanner [58].

Finally, Heintze et al. reported a global workflow accuracy in the range of ±10–15 µm, with replicas scanned with a laser [44]. In fact, the workflow accuracy was considered to be within the range of 15–20 µm by different research teams, using various profilometers and matching software [41,53,59]. Only Chadwick and Mitchell reported an accuracy of 4.4 µm, with the digitalization of a metal-coated impression and the use of a non-contact electrical gauge profilometer [60].

3.5. Matching software (MS) setting procedure

Twenty-four of 34 studies on digital profilometry reported setting details of the MS used. The most often used MS was Match 3D software (17 studies), whereas Ansur Software (Regents, University of Minnesota, Minneapolis, MN) was used in five studies in the 1990s. Other marketed or homemade software programs were also reported but in only one study for each of them. The superimposition of the two images to be compared was obtained by performing numerous iterations (≈8000) of combined rotations and translations of the images in the areas that were less subjected to changes. Some authors used reference areas, which were determined on buccal or lingual surfaces, or on occlusal surfaces free of occlusal contact points, as detected on intraoral pictures [42,44,49,61], while the others set the software to eliminate, during the matching process, points that exhibited a vertical

measurement difference superior to a predefined threshold. Those points were further re-included for the final wear analysis [55,62]. Another approach was to perform a first rough alignment to identify points without vertical measurement difference, which served as references to perform a second and finer alignment [63].

The best fit was obtained when the minimum vertical root mean square (RMS) was reached on 1000 or 1200 points at least, which varied between 15 and 30 µm, depending on the study [46,62,64–66]. Schmid-Schwap et al. set the limit at 20 µm for anterior teeth and 30 µm for molars, which are more difficult to superimpose [42]. If the conditions were not respected, data were excluded from the studies. Heintze et al. and Stober et al. added another control related to z-value distribution in slightly worn areas [5,44], in which superimposition most often resulted in negative but also positive values, related to workflow inaccuracies. They considered it a successful match when they obtained a symmetrical distribution of those values around zero (±15 µm); if those conditions were not respected, the data were also excluded from the study.

3.6. Positive control

In 7 studies, the authors monitored enamel wear in parallel with the studied materiel. Enamel was used as a positive control to validate the measurement method. This control was based on the work of Lambrechts et al. in 1989 [67], who stated that the enamel wear rate should be around 29 µm per year for molars and 15 µm for premolars.

3.7. Unusable data

Wear studies encountered the same difficulties as other clinical studies regarding patient drop-out. Incorrect impressions, unreliable scans, unmatchable

casts, and unexpected occlusal adjustments constitute additional sources of unusable data. Stober et al. [68], Heintze et al. [44], Ohlmann et al. [69], and Schmid-Schwap et al. [42] reported a total of 14%, 25%, 29%, and 31%, respectively, of unusable data (occlusal adjustments, waste impression, unusable scan, and patient drop-out). Bartlett et al. also reported 5% of unusable scans [34]. Authors who used reference elements such as disc metallic indexes [34] or pits [66] reported a high rate of unusable data due to the instability (81%–87% of discs were reported to be lost) or to the wear of those indexes that were bonded on the teeth.

3.8. Wear measurement results (Table 5.2)

For composite materials, mean vertical height loss was shown to vary between 27 µm after 5 years [45] and 102 µm after 1 year [66]; many authors reported values between 20 and 40 µm after 1 year [15,41,52]. For resin-based denture tooth materials, wear measurement protocols used were very similar [5,64,68]. The maximum vertical height loss varied between 140 µm and 225 µm after one year [5,64,68], composites appearing to be more resistant to wear than pure PMMA resins. Ceramic material mean vertical height loss was reported to vary between 12 µm [70] and 116 µm per year [54] for feldspathic ceramic, between 10 µm per 6 months [62] and 127 µm per year [57] for zirconia and between 78 µm after 4 years [56] and 148 µm after 1 year [54] for lithium disilicate glass-ceramics. Enamel mean vertical height loss was often reported around 10 µm per year [57,61,62], this value being used as a reference.

Finally, wear was shown to be more pronounced on molars than on premolars [57,71]. All authors reported wide variations related to the patient.

The different key-steps of the wear measurement workflow in clinical studies are summarized in Fig. 5.2, along with the main parameters influencing measurement accuracy and precision.

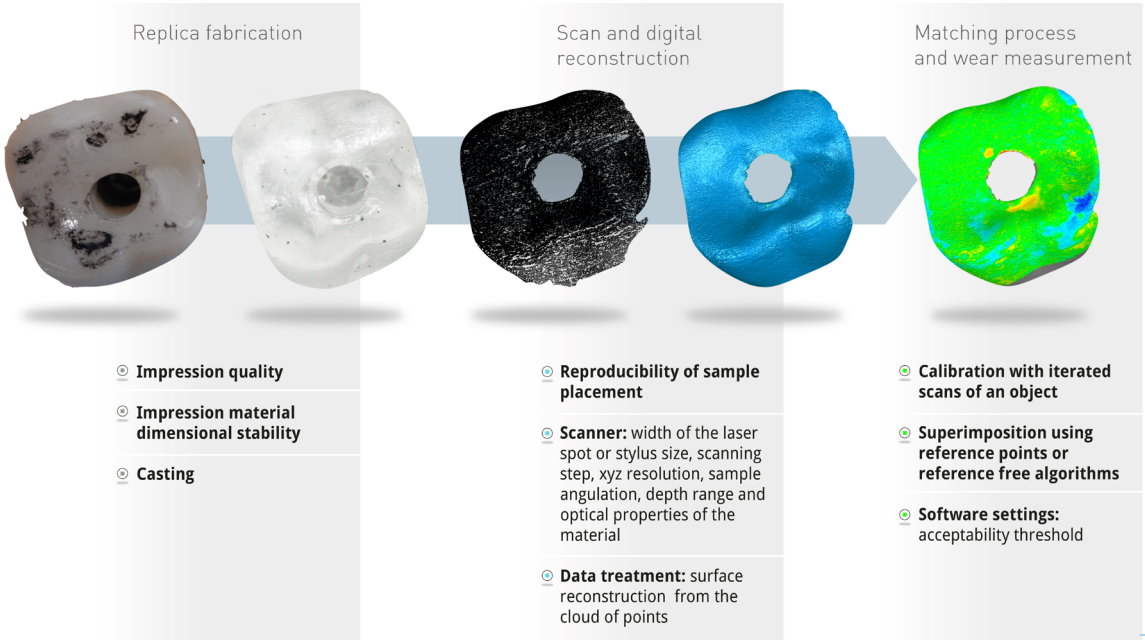

Fig 5.2 *Schematic representation of wear measurement workflow with main parameters influencing accuracy. Images refer to the occlusal face of a full zirconia screw-retained implant restoration on a molar.*

4. DISCUSSION

The global inaccuracy and imprecision of a wear measurement workflow is the sum of the errors of each step of the procedure taken independently. To be relevant, the accuracy and precision of a system should be significantly inferior to the minimum amount of wear that is expected in a specific clinical situation (around one tenth of this value) [21]. Consequently, with a reported mean global accuracy of 15–20 µm, the current wear measurement methods are relevant tools to detect changes of approximately 100 µm [21], that is, for a study of denture teeth wear, for example; however, a higher performance is needed to study dental tissues and restorative-material wear with respect to

wear values reported in the literature. In the present review, the inconsistency of reported values (for example, zirconia was reported to submit more to wear than enamel) confirms the unreliability of measurement workflows.

Currently, digital profilometry combined with MS is reputed as the best technique for wear measurement in clinical studies [21,72,73] and is the most often used. Yet the purchase of a 3D scan and matching software remains expensive [72]. Regarding a mechanical profilometer, it must be noted that its resolution is strongly related to its curvature radius, which can encounter small defects in its penetration and significantly reduce resolution. Moreover, mechanical profilometers present a significantly slower acquisition rate due the point-by-point acquisition procedure, whereas optical scanners acquire the image by full lines or areas, reducing acquisition time to approximately 30 min [19,59]. Finally, a mechanical stylus bends beyond 45°, which constitutes an issue regarding cusp angulation [27]. Stereomicroscopy equipped with stereophotogrammetry, which was described in the 1980s and used successfully in some *in vitro* studies [74,75], has never been employed for clinical research. Interferometry was also used *in vitro* but still not *in vivo* [76,77]. In fact, the issue with interferometry is its lateral resolution, which decreases when the vertical resolution increases.

As a result of the present review, some recommendations can be formulated to improve and standardize wear measurement:

1 Replica use should be avoided as much as possible. This option is valid for removable dentures or screw-retained implant restorations, which can be removed and directly scanned. In that objective, implant replicas can be used in the scanning device to improve repositioning [70]. The use of intraoral scanners is promising for the study of wear of restorative materials and tooth tissues [5,78–81], but at the present time they show an accuracy and precision around a maximum of 30 µm in *in vitro* conditions [82], which is not sufficient. Consequently, some evolutions are still necessary before recommending their use for quantitative wear measurement.

2 Measurement systems should be systematically calibrated to evaluate reliability, reproducibility, and repeatability of the scanning procedure, sample repositioning, and superimposition procedure, respectively [58]. The calibration procedure involves four steps: iterated superimposition of identical scans (superimposition process), superimposition of iterated scans of a standard model without displacing it from the positioning device (acquisition), superimposition of iterated scans of the standard model after removal and replacement in the positioning device (acquisition + superimposition), and, finally, superimposition of scans of duplicates of the standard model (complete procedure, including impression and casting).

3 Positive control should be systematically used, when possible.

4 Last-generation optical scanners should be preferred to contact gauge scanners. It must be noted that the reliability of scanners was shown to decrease when the sample presents an important angulation or cusp slope (>60°) [19,27]. Indeed, the projected light spots are too distorted for a correct capture of the return signal by sensors [59]. Major improvement in OPD could come from the development of laser triangulation systems coupled with a confocal microscope, which provides laser spots with reduced dimensions and then a better resolution [83]. Yet measurements on translucent materials such as ceramics are still a problem regarding diffusion phenomena. Other technologies, such as accordion fringe interferometry (AFI) and conoscopic holography, may provide satisfying resolution [84] but are not yet documented in clinical studies. However, if structured-light scanning systems could improve vertical resolution and acquisition speed, they would show a limited depth range, which can constitute an issue regarding the analysis of occlusal anatomy.

5 The scanning step should be at least inferior to the xy resolution (i.e., 20–25 µm) for the scanning to cover the whole surface. Accuracy and precision could even be significantly improved with a lower scanning step, but this change

would dramatically increase the acquisition time. Rodriguez et al. evaluated the acquisition time to 40 min for a 15mm×15mm surface, using a 50 µm step, and to 210 min with a 15 µm-step [59]. Consequently, a good compromise between experimental time and precision could be defined at around 20 and 25 µm.

6 For the matching procedure, the use of reference areas should be avoided in favor of the use of software settings (elimination of points that exhibit a vertical measurement difference superior to a predefined threshold), which allow for a better superimposition. Indeed, they eliminate the risk of operator-dependent imprecision related to the reproducibility of sample placement in the holder and to definition of the reference points at the different evaluation times. Also, the use of indexes bonded to teeth should be avoided because of the risk of loss or wear [34,66]. The definition of acceptability thresholds, related to the root mean square values, for example (which should be inferior to 20 or 30 µm), are also meaningful to ensure the quality of the superimposition process [42,46,62,64–66]. It is the same for the control of the distribution of the z-value in preserved areas (which should be around 15 µm) [5,44].

7 Regarding the type of analysis, 2D or 3D can be used since there is no proof that one technique is better than the other.

8 The large amount of unusable data reported in analyzed studies [42,44,68] (unreliable scans, unsuccessful matching, patients dropping out) should be taken into account in sample size calculation.

Finally, with respect to the current reliability of clinical wear measurement of restorative materials, *in vitro* studies, despite their limitations, are still interesting screening tools to compare different materials.

5. CONCLUSION

This systematic review showed an important lack of standardization of wear measurement procedures in clinical studies and an important variation in terms of performance of the different measurement workflows described. Moreover, methods reported a 10–15 µm global accuracy, which is not sufficient to analyze short-term wear of restorative materials and tooth tissues. The present review introduces some guidelines to standardize and improve the performance of measurement workflows, notably limiting the use of replicas, using standardized calibration procedures and positive controls, optimizing the settings of scanners and matching protocols, and taking into account unusable data. Future perspectives for clinical wear measurement include the evolution of intraoral scanners, which should avoid the use of replicas and improve final accuracy and precision.

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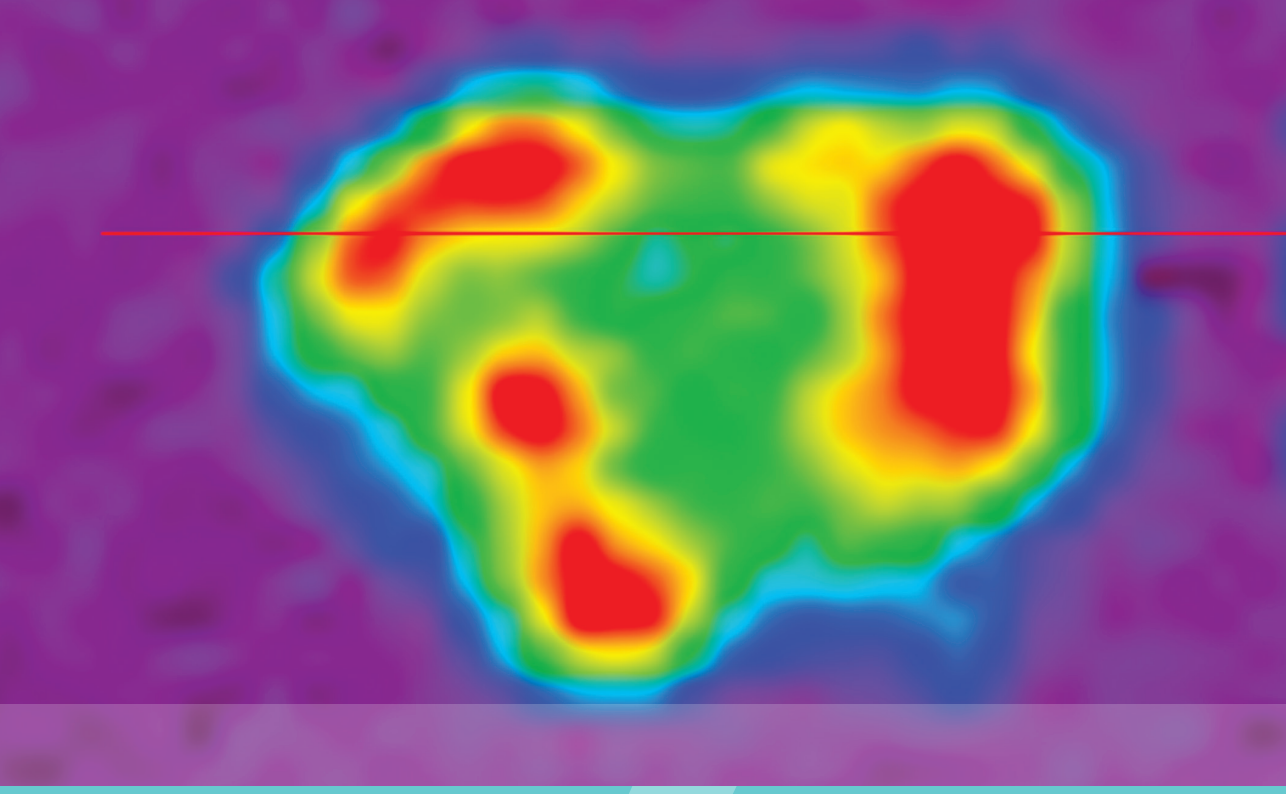

Intraoral low-temperature degradation of monolithic zirconia dental prostheses: Results of a prospective clinical study with *ex vivo* monitoring

Vinciane Koenig[1,2]
Sandrine Bekaert[1,2]
Nathalie Dupont[1,2]
Alain Vanheusden[1,2]
Stéphane Le Goff[3]
Thierry Douillard[4]
Jérôme Chevalier[4]
Nadia Djaker[5]
Marc Lamy de la Chapelle[6]
Frédéric Amiard[6]
Nadia Dardenne[7]
Claudine Wulfman[1,3,a]
Amélie Mainjot[1,2,a]

[1] *Dental Biomaterials Research Unit (d-BRU), University of Liège (ULiège), 45 Quai G. Kurth, Liège, 4020, Belgium*
[2] *Department of Fixed Prosthodontics, Institute of Dentistry, University of Liège Hospital (CHU), 45 Quai G. Kurth, Liège, 4020, Belgium*
[3] *Unité de Recherches en Biomatériaux Innovants et Interfaces (URB2i) – EA4462, Faculté de Chirurgie Dentaire, Université Paris Descartes, Sorbonne Paris-Cité, Montrouge, 92120, France*
[4] *Université de Lyon, INSA Lyon, CNRS, MATEIS, UMR 5510, F-69621 Villeurbanne, France*
[5] *Université Sorbonne Paris Nord, CNRS, UMR 7244, CSPBAT, Laboratoire de Chimie, Structures et Propriétés de Biomatériaux et d'Agents Thérapeutiques, 93017 Bobigny, France*
[6] *Institut des Molécules et Matériaux du Mans (IMMM - UMR6283), Université du Mans, avenue Olivier Messiaen, 72085 Le Mans Cedex 9, France*
[7] *Department of Public Health, University of Liège, 4000 Liège, Belgium*
[a] *These authors contributed equally.*

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ABSTRACT

Objective: To investigate the intraoral development and kinetics of low-temperature degradation (LTD) in second-generation 3 mol.% yttria-doped tetragonal zirconia polycrystal (3Y-TZP) monolithic prostheses, as well as the influence of masticatory mechanical stress and glaze layer on it.

Methods: A total of 101 posterior tooth elements were included in a prospective clinical study, which included *ex vivo* LTD monitoring (at baseline, 6 months, 1 year, and 2 years) using Raman spectroscopy (n = 2640 monoclinic phase measurement points per evaluation time) and SEM. Four types of areas (1–2 mm^2 surface, 6 on molars, and 4 on premolars) were analyzed on each element surface: occlusal, axial, glazed, or unglazed. Raman depth mapping and high-resolution SEM were performed on the selected samples.

Results: LTD developed in 3Y-TZP monolithic restorations 6 months after intraoral placement and progressed with time. After two years, the tetragonal-to-monoclinic transformation was non-uniform, with the presence of localized clusters of transformed grains. In axial areas, the grain aspect was typical of the classical nucleation-growth process reported for LTD, which progresses from the surface to a depth of several tens of microns. However, in occlusal areas, tribological stress generated surface crushing and grain pull-out from the clusters, which induced an underestimation of the aging process when the evaluation was limited to monoclinic phase quantification. Glazing cannot be considered a protection against LTD.

Significance: If LTD occurs in dental prostheses in the same way as in orthopedic prostheses, its clinical impact is unknown and needs to be further studied.

Keywords: 3Y-TZP - Computer-aided design/computer-aided manufacturing - Raman spectroscopy - *t-m* transformation - Aging - Biomaterials

1. INTRODUCTION

A low-temperature degradation (LTD) of a 3 mol.% yttria-doped tetragonal zirconia polycrystal (3Y-TZP) prosthesis was described for the first time in 1981 [1]. This aging phenomenon is characterized by a slow surface transformation to the stable monoclinic phase in the presence of water within a certain temperature range typically from room temperature to approximately 400 °C. Transformation initially starts in the isolated grains on the surface by a corrosion stress mechanism and propagates internally in a slow nucleation and growth process [2]. Nucleation on a particular grain at the surface in contact with water leads to a volume increase, which induces stress on neighboring grains and triggers their transformation and the formation of microcracks. This offers a path for water to penetrate by capillarity, allowing the process to also occur in the underlying layers [2]. This phenomenon depends on several factors, such as the grain size, the amount and distribution of stabilizers, and the presence of manufacturing residual stresses [3]. The LTD of 3Y-TZP ceramics used in orthopedic devices has catastrophic clinical consequences. In the early 2000s, fractures of approximately 600–800 zirconia heads within two years after implantation were reported. Seven batches of the Prozyr® material, manufactured with tunnel furnaces and expected to have increased sensitivity to LTD, were used [4]. Several studies on LTD have been conducted at the laboratory [5–11] and a few *in vivo* studies on aging, using retrospective explant analyses, have confirmed some of the features observed at the laboratory and showed surface degradation of retrieved zirconia hip implants [4,12–16] and/or related strong osteolysis [17–19]. LTD accounted for some clinical failures related to the use of zirconia in orthopedics: the Prozyr® heads broke because of crack propagation from the micro-cracked transformed region loaded in tension, and some retrieved heads showed extensive grain pull-out, which led to material debris, chronic inflammation, and osteolysis in the worst cases [6]. Those issues, especially the "Prozyr® failures", had a catastrophic impact on the use of Y-TZP in the field of orthopedics, in spite of the contrasting results for different zirconia heads, which were good and showed low wear rates [20,21].

Concomitant with its abandonment in orthopedics, the use of Y-TZP has increased in the dental field because of its better optical and biocompatibility properties when compared with metal alloys and its high mechanical resistance when compared with glass-ceramics. Surprisingly, 3Y-TZP is extremely popular and widely used in the field of dental prostheses [22] ; however, LTD has been sparsely investigated *in vivo* during clinical studies. The mechanisms of LTD have been relatively well-described, but there are still debates on the *in vivo* performance of Y-TZP in dental applications, particularly the 3Y-TZP second generation called the "translucent" zirconia [23]. The increased translucency of these materials allows for the fabrication of high-strength monolithic crowns and fixed dental prostheses (FDPs), which are not needed in the posterior zone, to be covered by a feldspathic ceramic layer to obtain satisfactory esthetics. However, translucency is generally improved through a decrease in the content of alumina (a *t-phase* stabilizer) and/or an increase in the sintering temperature to reduce porosity and increase the grain size (2 h sintering at 1350 °C gives a 0.3 µm grain size, while it is higher than 2 µm at 1650 °C), which, in turn, increases the material metastability and LTD sensitivity [23,24] (Table 6.1). Owing to the high variability of the materials tested, protocols employed, and the risks of bias of several studies [25], the results for the influence of LTD on material strength are controversial ; some studies have shown that LTD significantly decreases [5,26–28] or increases [29,30] the flexural strength of various Y-TZP dental ceramics, while others have reported that it does not have any influence [31–37]. However, according to a meta-analysis of *in vitro* studies published in 2015, LTD induced by autoclave aging (aging duration longer than 20 h, pressure greater than or equal to 2 bars, and temperature of 134 °C) significantly increases the *m-phase* content and decreases the flexural strength of first- and second-generation 3Y-TZP dental ceramics [25]. Some studies reported that LTD was responsible for a significant decrease in flexural strength when 50% of the sample surface was transformed [25–27] and recent international standard rules for zirconia [38] (ISO 13356- 2015) state that the monoclinic phase content should not exceed a maximum of 25% for Y-TZP implants to be

considered suitable for biomedical purposes after aging in an autoclave at 134 °C and 2 bars for 5 h. However, *in vitro* autoclave aging conditions are different from the body environment [39]. Moreover, the exposure to saliva (an ionic solution), acidic beverages, and constant changes in pH have not

Zirconia	Composition	Properties	Indications
First-generation	3Y-TZP (<15% cubic phase)	High opacity/ refractive index High strength & toughness 1000 - 1500 MPa flexural strength (manufacturers' data)	Framework for veneered restorations
Second-generation : Translucent	3Y-TZP (<15% cubic phase) ↘ Alumina Sintering at higher temperature to reduce porosities ↗ Metastability	↗ Translucency ↗ LTD 900 - 1300 MPa flexural strength (manufacturers' data)	Monolithic (or veneered) posterior restorations
Third-generation: High-translucent	4Y-PSZ (>25% cubic phase) 5Y-PSZ (>50% cubic phase) Grain size: 1.5 µm Metastability	↗ Translucency ↘ LTD ↘ Strength & toughness 400 - 1000 MPa flexural strength (manufacturers' data)	Monolithic (or veneered) single-unit restorations Max 3 elements-bridges

Table 6.1 *Characteristics of the current generation of dental zirconia materials in terms of composition, properties, and indications.*

been taken into account [31,40], and mechanical stress due to masticatory forces (notably tribological effects) has not been considered, which can accelerate Y-TZP degradation, as shown *in vitro* [41] and *in vivo* for zirconia hip prostheses (explant analysis) [7,15]. Recently, Miragaya et al. [42] and Schepke et al. [43] reported *in vivo* data on the LTD of Y-TZP dental ceramics, but they were limited. In the first study, flat Y-TZP specimens were attached to personalized intraoral resin appliances and exposed to the oral cavity (but not tomechanical stress) for two months, whereas in the second study, the transgingival part of zirconia implant abutments was observed after one year of clinical function.

In conclusion, the rationale for the long-term follow-up on dental prostheses is represented by the following questions: Is there aging in current dental zirconia? Are masticatory forces/contact stresses important in the process? If there is an operant, is this a clinical issue? Therefore, an original clinical research protocol, which includes *ex vivo* monitoring, was designed to investigate the *in vivo* development and kinetics of LTD in second-generation zirconia dental prostheses and the influence of masticatory mechanical stress on this process over 5 years [44]. The secondary objective includes the influence of glaze protection on this process. The present work reports the 2-year results of this 5-year prospective clinical study on intraoral LTD. Additionally, this project also analyzed the general clinical performance (notably evaluation using criteria of the World Dental Federation) and material wear of prostheses using 3D-profilometry: the 2-year results of these specific investigations were reported in a previous paper [45].

2. MATERIALS AND METHODS

2.1. Study design

The protocol of this prospective clinical study was approved by the Ethics Committee of the University Hospital Center (CHU) of Liege and was registered in the ClinicalTrials.gov database (Identifier NCT02150226). The study design is illustrated in Fig. 6.1. Written patient consent was obtained before inclusion. The patients were treated by four experienced operators in the Department of Fixed Prosthodontics, Institute of Dentistry, CHU of Liege, Belgium. The eligibility criteria included the need for a molar or premolar crown (maximum of 6 elements per patient). The restorations were realized either on the teeth or the implants, and FDPs were also included, provided they were on the implants and limited to three elements (maximum 2 FDPs per patient). The exclusion criteria were severe or acute periodontal or carious diseases and poor oral hygiene. Patients without antagonistic teeth or those with removable prostheses as antagonists were excluded. A total of 47 patients, with a mean age of 54.3 ± 15.3 years, were recruited (14 males, 33 females). A total of 75 restorations (101 tooth elements) were included. Sample descriptions of patients, restorations, and tooth elements are presented in Table 6.2. The participants received no financial compensation, although the treatment and prostheses were free of charge. All of the patient-related data were recorded in a specifically designed online database, to facilitate easy access to clinical data, pictures, and data obtained from *ex vivo* analyses for consultation. The database is hosted on a university hospital-secured server.

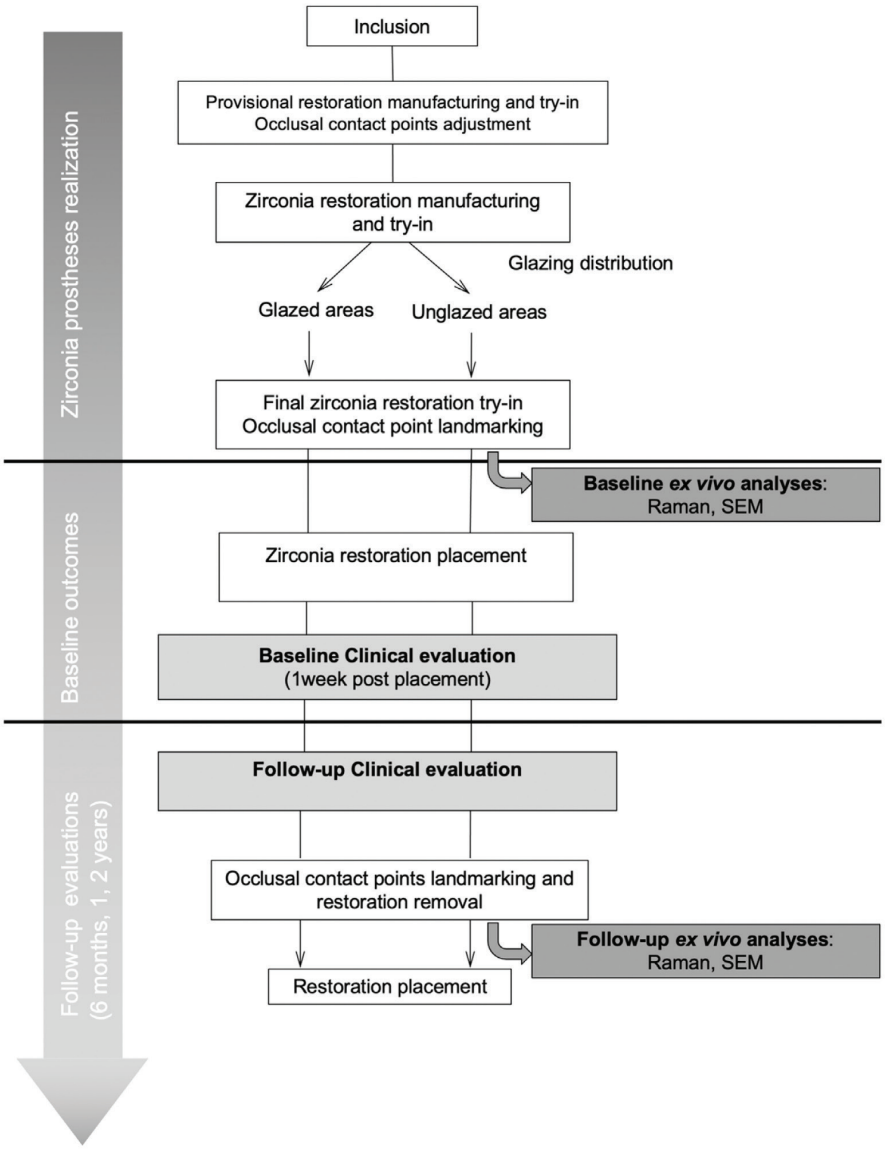


Fig 6.1 *Study design.*

2.2. Clinical procedure and fabrication of the restorations

2.2.1. Tooth preparation and impression for tooth or implant-supported prostheses

All clinical and technical procedures were performed strictly according to the manufacturer's recommendations. The teeth were prepared following standardized criteria (1.0–1.5 mm occlusal depth cut to achieve the appropriate occlusal anatomy, 1.0–1.5 mm functional cusp tip reduction, 0.5 mm gingival chamfer reduction, and a 6–8 degree taper to the axial walls). A double-mix impression was performed with high- and low-viscosity A-silicone impression materials (Aquasil Heavy/XLV, Dentsply De Trey, Konstanz, Germany). An open tray impression technique was performed using the same impression material used for implant restorations. Two implant systems (NobelReplace, Nobel Biocare, Gothenburg, Sweden ; and Standard Implants, Straumann, Basel,Switzerland) were used in this clinical study. The shade was registered using the Vita Classic System (Vita Zahnfabrik, Bad Säckingen, Germany). Restorations of antagonistic teeth were replaced in the presence of a deficient marginal joint, decay or inadequate morphology.

2.2.2. Provisional restorations

Before manufacturing the zirconia restorations, CAD-CAM composite provisional crowns (Lava Ultimate, 3M ESPE, Seefeld, Germany) or PMMA-provisional FDPs were made. After die scanning the restoration was designed using CAD/CAM software: Exocad (Darmstadt, Germany) or Dental Wings (Montreal, Canada) (DPI Lava milling center, Anderlecht, Belgium). Specific buccal and palatal undercuts were added to the crown design to facilitate cemented crown removal (Fig. 6.2a). The file was transferred to a milling machine for manufacturing (Lava CNC 500, Serial Number: 07019 (2009), 3M ESPE). The provisional restorations were adjusted in the mouth and used as a mock-up for the design of zirconia restorations. Particular attention was

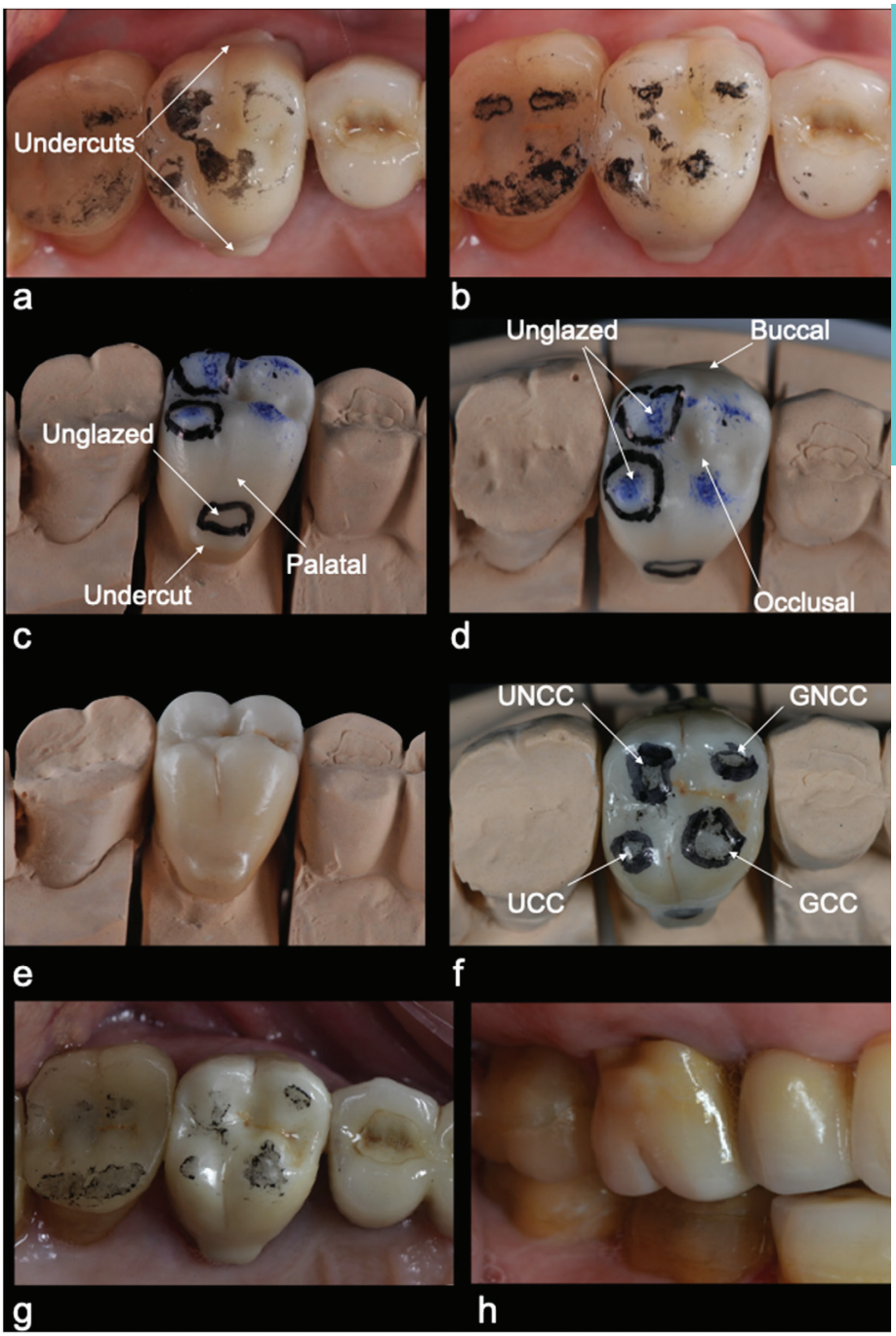

Undercuts
a
b
Unglazed
Buccal
Unglazed
Palatal
Undercut
Occlusal
c
d
UNCC
GNCC
UCC
GCC
e
f
g
h

Fig 6.2 *Realization of a cemented zirconia crown (tooth #16).*
(a) Occlusal contact points before adjustment on the Lava Ultimate provisional crown.
(b) Occlusal contact points after adjustment to obtain 1–2 mm^2 flat surfaces on each cusp. The provisional crown was scanned and used as a mock-up for the zirconia crown.
(c) Lava Plus zirconia crown after intraoral try-in and registering of occlusal contact points. Areas that will not be glazed are landmarked with permanent ink. The palatal surface is left unglazed on a part above the undercut.
(d) For molars, two cusps have been randomly selected to remain unglazed: one centric cusp (unglazed centric cusp; UCC) and one non-centric (unglazed non-centric cusp; UNCC). The two other cusps were called "glazed centric cusp" (GCC) and "glazed non-centric cusp" (GNCC).
(e) Glazed Lava Plus zirconia final crown.
(f) Landmarking of areas to be analyzed after intraoral try-in: occlusal contact points and the axial areas on the buccal and palatal surfaces located up to the undercut created to remove the crown.
(g) Intraoral occlusal view after baseline evaluation and restoration placement.
(h) Buccal view.

paid to the occlusal contact point adjustment to obtain at least one flat contact surface of a minimum of 1 - 2 mm^2 per cusp by grinding or adding a composite (Fig. 6.2a and b).

2.2.3. Zirconia prostheses

Provisional restorations were scanned for zirconia restoration fabrication (Lava Plus, 3M ESPE, Seefeld, Germany) with the same milling system. Lava Plus is a second-generation 3Y-TZP, with a higher translucency than Lava Frame Zirconia (first-generation) due to a lower alumina content of 0.1%, optimally distributed within the material (Lava Plus, technical product profile, 3M). Sintering was performed according to the manufacturer's instructions at 1450 °C for 2 h. The zirconia restorations were polished with a sequence of three diamond-impregnated silicone discs (Edenta AG, Hauptstrasse, Switzerland) and a diamond polishing paste (Bredent, Senden, Germany). Implant-supported restorations were bonded onto a specific titanium abutment (1000er-Serie, Medentika, Hugelsheim, Germany) with a resin composite cement (RelyX Ultimate [3M, Seefeld, Germany] in the first part of the study or Multilink Hybrid Abutment [Ivoclar Vivadent, Schaan, Liechtenstein] in the second part) after sandblasting the abutment and the zirconia restoration with 50 µm alumina particles at 2 bar according to the manufacturers' recommendations.

Zirconia restorations were tried-in, and, if needed, occlusal contact points were adjusted with a diamond bur (ISO 524 grit size) and polished using a sequence of three zirconia-dedicated silicon gums (Diasynt Plus/Diacera Zirconium, Eve Ernst Vetter, Pforzheim, Germany), following the manufacturer's recommendations. The need for occlusal adjustments was encoded in an online database for each occlusal area.

For restorations on implants that had no undercuts on buccal and lingual/palatal surfaces, a small groove was designed with a diamond bur (Fig. 6.3a) as a landmark for *ex vivo* analyses of axial surfaces, which were the control areas (free of mechanical stress). For molars, two cusps were randomly selected to remain unglazed: one centric cusp (UCC) and one non-centric cusp (UNCC). The two other cusps were called "glazed centric cusp" (GCC) and "glazed non-centric cusp" (GNCC) (Fig. 6.2c and d). For premolars, one cusp was randomly selected to remain unglazed (Fig. 6.3a). The buccal surface was systematically glazed, and the lingual/palatal surface was left unglazed on a part above the undercut or groove (Figs. 6.2c and 6.3a). The glaze (IPS empress stains and eMax Ceram glaze, Ivoclar Vivadent, Schaan, Liechtenstein) was sintered at 780 °C for 1 min. Definitive bonding (bond was eliminated during glaze firing) on the specific titanium abutment was performed following a previously described procedure. The glazed restorations were tried-in, and occlusal contact point areas (one per cusp, i.e., four on molars and two on premolars), as well as lingual/palatal and buccal areas, were marked for *ex vivo* analyses and registered with a picture (Figs. 6.2f and 6.3b).

2.2.4. Zirconia prostheses placement and removal for *ex vivo* analyses

Baseline *ex vivo* analyses of the zirconia restorations were performed before placement. Screw-retained restorations were torqued at 35 Ncm^{-1} (Fig. 6.3c and d). To remove the gold sputter coating, the restorations were cleaned first with an alcohol-soaked cotton ball, brushed with tooth brush and tooth paste, and finally immersed in an ultrasonic bath containing alcohol for 3 min.

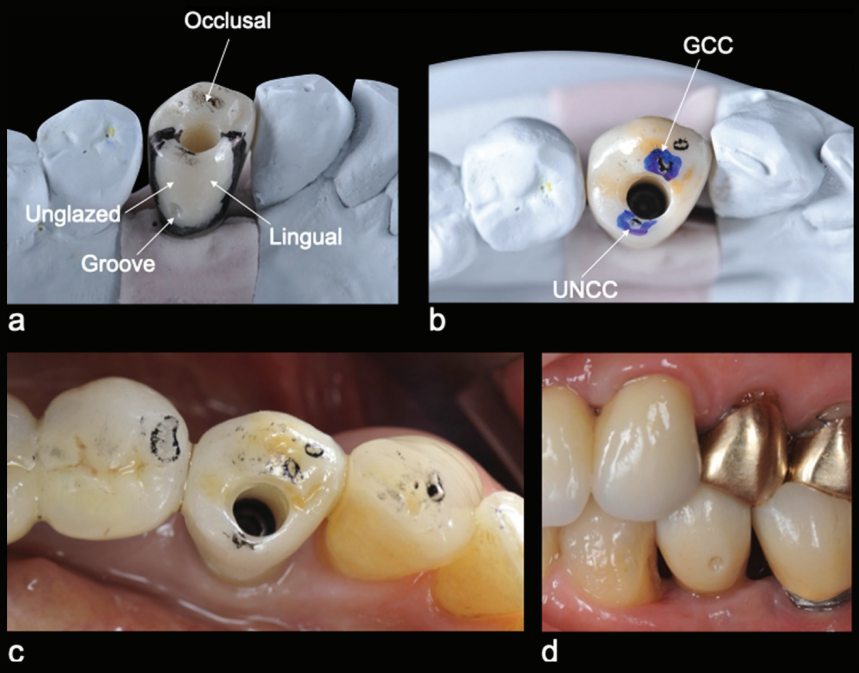


Fig 6.3 *Screw-retained zirconia crown on an implant (tooth #34), which was realized in the same way as the cemented crown in Fig. 6.2.*
(a) Lava Plus zirconia crown after intraoral try-in and registering of occlusal contact points. Areas that will not be glazed are landmarked with permanent ink. The lingual surface is left unglazed on a part above the groove. For premolars, one cusp is randomly selected to remain unglazed.
(b) Landmarking of the areas to be analyzed after intraoral try-in of the final zirconia crown: occlusal contact points and the axial areas on the buccal and lingual surfaces are located up to the groove performed on the restoration surface.
(c) Intraoral occlusal view after baseline evaluation and restoration placement.
(d) Buccal view.

Cemented restorations were sealed with eugenol-free cement (RelyX Temp NE, 3 M ESPE) (Fig. 6.2g and h), and the teeth were disinfected with 2% chlorhexidine. After 6 months, the restorations were removed for *ex vivo* analyses. Provisional restorations replaced the zirconia restorations during the analyses. The zirconia restorations were placed again in the mouth of the patient, following the same procedure as for the first time. Evaluations were repeated after a one-year intraoral stay and after two years. The occlusal

contact points were landmarked with bite registration paper before removing the prostheses and encircled with permanent ink (Figs. 6.2f and 6.3b).

2.3. *Ex vivo* analyses

Ex vivo analyses included (1) phase transformation with Raman spectroscopy and (2) glaze wear using scanning electron microscopy (SEM). Sample descriptions of the *ex vivo* analyzed areas (occlusal or axial, glazed, or not glazed) (*n* tot = 528) are presented in Table 6.2.

2.3.1. Raman spectroscopy

Five measurement points (MPs) were randomly chosen for each occlusal contact area and each axial area (i.e., 30 measurements for a molar and 20 measurements for a premolar). Consequently, the analyses were performed for 2640 MPs at each evaluation time (baseline, 6 months, 1 year, and 2 years). Sample descriptions of the MPs are presented in Table 6.2.

Raman spectra were recorded using a Raman spectrometer (Horiba Jobin Yvon, Kyoto, Japan). The excitation laser was provided by a HeNe laser (632 nm), with 1 mW power focused at the surface of the specimen, and the Raman spectra were acquired using a charge-coupled device detector (Horiba-Jobin Yvon, Kyoto, Japan) with a cm^{-1} spectral resolution of 1 (1800 grooves/mm grating). The Raman spectrometer was combined with an optical microscope (Olympus LX71 ; Olympus Corporation, Tokyo, Japan). A confocal pinhole with adjustable diameter was used for confocal detection, and an 80× objective (numerical aperture 0.75) was used to achieve a 1 μm^3 resolution (lateral × axial). A pinhole aperture of 1.99 µm was used to reach a collection depth of 1.44 µm. The depth was determined using the calibration-wedge method [46]. The collected spectra were analyzed, and the transformed monoclinic volume fraction (V_{fm}), which is a measurement of the transformation volume ratio in the confocally probed

Patients (n tot = 47)	**% (n)**
Sex	
Female	70.2 (33)
Male	29.8 (14)
Restorations (n tot = 75)	**% (n)**
Crowns	
Implant screw-retained crowns	58.7 (44)
Implant cemented crowns	5.3 (4)
Tooth cemented crowns	17,3 (13)
Bridges	
Screw-retained bridges on two implants	17.3 (13)
3-element	14.7 (11)
2-element	2.7 (2)
3-element cemented bridge on two implants	1.4 (1)
Tooth elements (n tot = 101)	**% (n)**
Support	
Tooth	12.9 (13)
Implant	87.1 (88)
Tooth type	
Premolar	38.6 (39)
Molar	61.4 (62)
Analyzed areas (n tot = 528)	**% (n)**
Occlusal (n = 326)	
GCC	26.4 (86)
UCC	23.9 (78)
GNCC	29.1 (95)
UNCC	20.6 (67)
Axial (n = 202)	
Buccal (Glazed)	50 (101)
Lingual/Palatal (Unglazed)	50 (101)
Raman measurement points (MPs) (n tot = 2640)	**% (n)**
Occlusal (n = 1630)	
GCC	26.4 (430)
UCC	23.9 (390)
GNCC	29.1 (475)
UNCC	20.6 (335)
Axial (n = 1010)	
Buccal (Glazed)	50 (505)
Lingual/Palatal (Unglazed)	50 (505)

Table 6.2 *Sample description in terms of patients, restorations, tooth-elements, ex vivo analyzed areas, and Raman measurement points (MPs). GCC: glazed centric cusps, UCC: unglazed centric cusps, GNCC: glazed non-centric cusps, UNCC: unglazed non-centric cusps.*

volume, was estimated using Eq. (1), which was initially proposed by Clarke and Adar [47]

$$V_{fm} = \frac{I_m^{178} + I_m^{189}}{0.33\left(I_t^{145} + I_t^{256}\right) + I_m^{178} + I_m^{189}}$$

where, I_m and I_t are the intensities of the peaks (wave numbers in the superscripts) of the monoclinic and tetragonal phases, respectively. The Raman peak positions and intensities were obtained by fitting the Raman spectra with Lorentzian curves using Origin 8 software (OriginLab, Northampton, MA, USA).

2.3.2. SEM observations

After the Raman spectroscopy experiments, the restorations were gold-coated and observed with a JSM-6400 scanning electron microscope (JEOL Ltd., Tokyo, Japan) to determine the glaze wear.

2.3.3. Additional experimentations on selected samples

Additional observations were carried out on selected samples to verify the hypotheses formulated after the analysis of the Raman spectroscopy and SEM results.

2.3.3.1. Raman mapping.

Raman spectroscopy mapping was performed on one crown just after its machining (baseline) and on two crowns after the 3-year follow-up. Raman mapping was performed using a Raman spectrometer (Apha300 Apyron, Witec, Germany) equipped with a confocal microscope. The material was excited with a HeNe laser (633 nm, laser power: 22 mW), and the Raman spectra were recorded in a backscattering configuration using a 20× microscope Zeiss objective with a numerical aperture of 0.50. The spatial

resolution was 771 nm in the focus plane (material surface) and 2.5 µm in the plane perpendicular to the focus plane (in-depth). A spectral resolution of 1.77 cm^{-1} was used to observe the different bands related to the zirconia phase transformation. For the surface characterization, the mapping of a surface area of 100 × 100 $µm^2$ with a step size of 3.33 µm corresponding to 33 × 33 points of measurement (0.8 s of accumulation for each point) was first conducted. When the *m* phase was detected, high-resolution mapping of an area of 25 × 25 $µm^2$ with a step size of 700 nm was performed to reach the spatial resolution limit. The depth profiles of the selected zones were conducted along one line at different depths with a step size of 1 µm.

For each map, the recorded signal (Raman signal + background) was integrated with the spectral range of 24 cm^{-1} centered at 184 cm^{-1} using the built-in Raman spectrometer software. The signal value was automatically transformed to a color scale using the software.

2.3.3.2. High-resolution SEM observations without gold coating.

One crown was examined in-depth with a high-performance SEM at the 3-year follow-up (Supra 55 V P ; Carl Zeiss Microscopy GmbH, Oberkochen, Germany). This instrument was equipped with a thermal field emission gun to provide a combination of high brightness and high resolution. Moreover the microscope was fitted with a variable-pressure secondary electron (VPSE) detector. This VPSE detector comprised a specific arrangement of detectors designed to obtain electron-similar imaging in a gaseous environment (up to 133 Pa of gaseous nitrogen), allowing the observation of non-conductive samples without the usual requirement of a metalized surface coating. Thus, high-resolution topographic SEM imaging was performed on the areas subjected to mechanical stress orthose not used for chewing, with an accelerating voltage of 10 kV and a pressure of 10 Pa without any coating.

2.4. Statistical analysis

2.4.1. Sample size

The sample size (N) was determined based on the following considerations. The statistical unit was the tooth element characterized by its maximum LTD value measured at each time point (baseline, 6 months, 1, 2, 3, 4, and 5 years). A V_{fm} value above 50% indicated treatment failure because LTD is expected to induce a significant decrease in material flexural strength above this threshold (see Introduction). LTD is expected to induce a significant decrease in material flexural strength when 50% of the sample surface shows a tetragonal to monoclinic (*t-m*) transformation [25]. The overall proportion (π) of such treatment failures was defined as the primary outcome measure of the study. The study rationale was to reject the proposed treatment for $\pi > 0.20$: more than 20% of the treatment failures over time. Assuming a significance level (α) of 1% (Bonferroni correction for multiple time testing), a power 1- β of 90%, a proportion π of at most 0.08 (margin 0.12), and a one-sided Z test for a binomial proportion of 0.20, a sample size (N) of 91 teeth was needed to detect > 20% of treatment failures at each data point collection. To account for correlations between teeth within subjects and study withdrawals, the sample size was increased to a minimum of 100 teeth.

2.4.2. Statistical methods

The collected data were expressed as mean ± standard deviation (mean ± SD) for quantitative variables and a frequency table for categorical variables. An analysis of the normal distribution of the data was performed using the Shapiro–Wilk test. The time evolution of the transformation was analyzed using a generalized mixed binomial model (GLMM). The time and group effects, as well as the interaction effect between time and group, were tested. Contrast analyses were also performed to test the differences between each time point. Owing to the presence of excessive zeros, the

transformation percentage over time was analyzed using a zero-inflated non-negative binomial regression for repeated measurements, globally, and according to the area. The effect of glaze, as well as grinding procedures for occlusal contact point adjustments, on the transformation with time was also analyzed using a GLMM, globally, and according to the type of area (occlusal or axial). In all the tests, variations were considered statistically significant when the *p*-value was ≤ 0.05. Data analysis was performed using GraphPad Prism (GraphPad Software, San Diego,CA), SAS (version 9.4) statistical package, and R version 3.6.1 with the glmmTMB package.

3. RESULTS

Five catastrophic failures, which led to restoration loss, were observed after 1 year: one crown fracture, one composite core fracture, one root fracture, and two implant losses. After 2 years, 1 patient with a screw-retained crown dropped out of the study. One screw-retained crown could not be removed after one year, and a second one could not be removed after two years ; therefore, they were not analyzed from those evaluation times. Moreover, one screw-retained crown had a minor chipping and seven cases of restoration debonding were registered (three provisionally cemented crowns and four screw-retained crowns, which debonded from the titanium base).

3.1. Raman spectroscopy

At baseline and 6 months, all transformation-positive areas presented only one positive MP (V_{fm} > 0%) on 5 (one MP corresponding to a collection probe of 1 µm cube, i.e., two or three zirconia grains on an analyzed area of 1–2 mm^2) (Table 6.3). The percentage of zero values decreased significantly with time ($p < 0.0001$). This explains the low mean V_{fm} values and the important standard deviations presented in Table 6.4.

	n transformation-positive areas/ n tot areas (%)	Transformation-positive areas, which on 5 MPs, show:			
		4 zero values n (%)	3 zero values n (%)	2 zero values n (%)	1 zero values n (%)
Global					
Baseline	21/528 (3.98)[a]	21 (100)	0 (0)	0 (0)	0 (0)
6 months	18/528 (3.41)[a]	18 (100)	0 (0)	0 (0)	0 (0)
1 year	49/496 (9.88)[b]	42 (85.71)	7 (14.29)	0 (0)	0 (0)
2 years	57/480 (11.88)[b]	46 (80,70)	7 (12.28)	3 (5.26)	1 (1.75)
Axial					
Baseline	2/202 (0.99)[c]	2 (100)	0 (0)	0 (0)	0 (0)
6 months	5/202 (2.48)[c]	5 (100)	0 (0)	0 (0)	0 (0)
1 year	26/190 (13.68)[d]	20 (76.92)	6 (23.08)	0 (0)	0 (0)
2 years	32/184 (17.39)[d]	25 (78.13)	5 (15.63)	1 (3.13)	1 (3.13)
Occlusal					
Baseline	19/326 (5.83)[e,f]	19 (100)	0 (0)	0 (0)	0 (0)
6 months	13/326 (3.99)[e]	13 (100)	0 (0)	0 (0)	0 (0)
1 year	23/306 (7.52)[e,f]	22 (95.65)	1 (4.35)	0 (0)	0 (0)
2 years	25/296 (8.45)[f]	21 (84.0)	2 (8.0)	2 (8.0)	0 (0)

Table 6.3 *Areas with at least one transformation-positive measurement point (MP+). Distribution of the number of MPs+ per transformation-positive area at each evaluation time and for each type of analyzed area (occlusal and axial). Different superscripts (within the column) denote that there were significant differences between the evaluation times. P-values are from a generalized linear mixed model with a level of significance of $p < 0.05$.*

Looking at the evolution of all MPs taken globally, including axial and occlusal MPs (*n* tot = 2640), the percentage of transformation-positive MPs (MPs+) and the mean V_{fm} significantly increased with the duration between baseline and 2 years ($p < 0.0001$ and $p = 0.012$, respectively) (Tables 6.4 and 6.5, Fig. 6.4a). However, the mean V_{fm} significantly decreased between baseline and 6 months ($p = 0.0036$) (Table 6.4). The percentage of MPs with a critical amount of the *m* phase (V_{fm}>50%) (MPs+50%) did not show any significant change between baseline and 2 years ($p = 0.12$) ; it significantly decreased between baseline and 6 months ($p = 0.034$) and increased between 6 months and 2 years ($p = 0.0068$) (Table 6.5, Fig. 6.4b).

		MPs (n)	Mean V_{fm} (%)	SD	Min	Max
Global	Baseline	2640	0.26[a]	4.32	0.00	90.00
	6 months	2640	0.12[b,c,d]	2.25	0.00	93.00
	1 year	2480	0.52[c,d]	5.19	0.00	96.00
	2 years	2400	0.62[d]	5.46	0.00	90.00
Axial	Baseline	1010	0.02[e]	0.41	0.00	12.00
	6 months	1010	0.06[e]	0.91	0.00	21.00
	1 year	950	0.87[e]	6.37	0.00	96.00
	2 years	920	1.22[e]	8.10	0.00	90.00
Occlusal	Baseline	1630	0.41[f]	5.48	0.00	90.00
	6 months	1630	0.15[f,g,h]	2.78	0.00	93.00
	1 year	1530	0.31[g,h]	4.28	0.00	93.00
	2 years	1480	0.25[h]	2.68	0.00	64.00

Table 6.4 *Results of Raman analyses: mean V_{fm} value with standard deviation and range (min—max), at each evaluation time and for each type of analyzed area (occlusal and axial). Different superscripts (within the column) denote that there were significant differences between the evaluation times. P-values are from a zero-inflated non-negative binomial regression for repeated measurements with the level of significance $p < 0.05$. MP: measurement point.*

		MPs n tot	MPs+ n (%)	MPs+50% n (%)
Global	Baseline	2640	21 (0.80)[a]	7 (0.27)[c,e,f]
	6 months	2640	18 (0.68)[a]	1 (0.04)[d]
	1 year	2480	56 (2.26)[b]	9 (0.36)[e,f]
	2 years	2400	73 (3.04)[b]	11 (0.46)[f]
Axial	Baseline	1010	2 (0.20)[g]	0 (0.0)[NA]
	6 months	1010	5 (0.50)[g]	0 (0.0)[NA]
	1 year	950	32 (3.37)[h]	6 (0.63)[NA]
	2 years	920	42 (4.57)[h]	8 (0.87)[NA]
Occlusal	Baseline	1630	19 (1.17)[i,j]	7 (0.43)[k]
	6 months	1630	13 (0.80)[i]	1 (0.06)[l]
	1 year	1530	24 (1.57)[i,j]	3 (0.20)[k,l]
	2 years	1480	31 (2.09)[j]	3 (0.20)[k,l]

Table 6.5 *Results of Raman analyses: number of measurement points (MPs), transformation-positive MPs with $V_{fm} > 0\%$ (MPs+), and transformation-positive MPs with $V_{fm} > 50\%$ (MPs+ 50%) at each evaluation time and for each type of analyzed areas (occlusal and axial). Different superscripts (within the column) denote that there were significant differences between the evaluation times. P-values are from a generalized linear mixed model with the level of significance of $p < 0.05$ and were not applicable (NA) for the axial MPs +50%.*

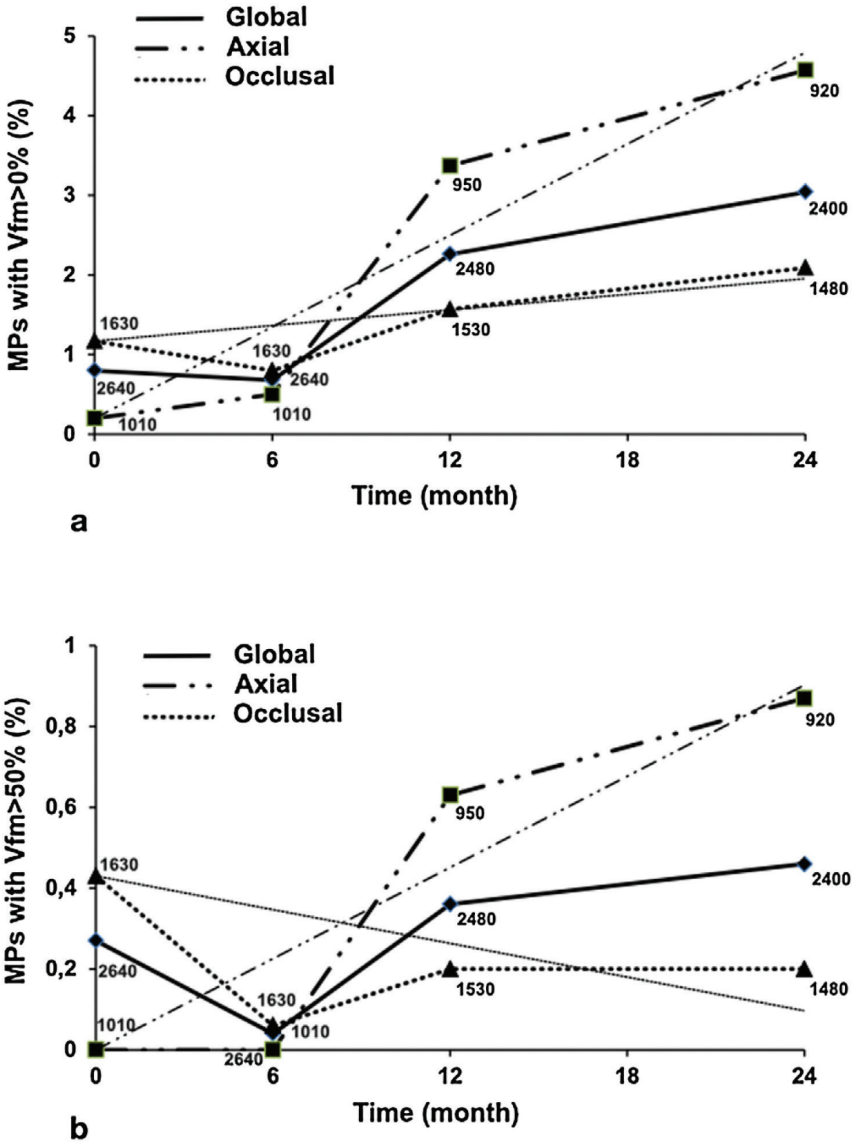


Fig 6.4 *(a) Time evolution of the percentage of measurement points (MPs) with a V_{fm} > 0% (MPs+) globally and according to the type of area (occlusal or axial).*
(b) Time evolution of the percentage of measurement points (MPs) with a V_{fm} > 50% (MPs+50%) globally and according to the type of area (occlusal or axial). In each figure, the global evolution in axial and occlusal areas is schematized by a straight line. Numbers indicate the total number of MPs at each evaluation time globally and according to the type of area (occlusal or axial).

The MPs + at baseline, particularly in occlusal areas, were significantly associated ($p = 0.0003$) with the use of a diamond bur to adjust the occlusal contacts with the antagonistic teeth during the zirconia restoration try-in (111 occlusal areas on 326, 34%, had to be adjusted).

There was no significant difference between the percentages of MPs + for the glazed and unglazed areas, considering the axial and occlusal areas taken globally and separately, irrespective of the evaluation time.

The results showed significant differences between the occlusal (subjected to mechanical stress) and axial (not subjected to mechanical stress) areas. The change in the percentage of MPs + and the mean percentage of V_{fm} with time varied with the type of area studied ($p = 0.0013$ and $p = 0.0016$, respectively).

In axial areas, the percentage of MPs + significantly increased with time, with a significant increase between 6 months and 2 years ($p < 0.0001$) (Table 6.5, Fig. 6.4a), while the mean V_{fm} remained stable (Table 6.4). Regarding the change in the percentage of MPs+50% over time, the analyses were not appropriate, given the absence of MPs+50% at baseline and 6 months (Table 6.5, Fig. 6.4b).

In occlusal areas, the percentage of MPs + remained stable between baseline and 2 years, with a significant increase between 6 months and 2 years ($p < 0.05$) (Table 6.5, Fig. 6.4a), while the mean V_{fm} significantly decreased with time, particularly between baseline and 1 and 2 years, respectively ($p = 0.027$ and $p = 0.0023$, respectively) (Table 6.4). The percentage of MPs + 50% was shown to significantly decrease between baseline and 6 months ($p = 0.034$) and it remained stable (Table 6.5, Fig. 6.4b).

3.2. SEM observations

The SEM results revealed that the glaze was worn out in most of the occlusal areas (70.2% of cusps) after 6 months and in all of them after 1 year (Fig. 6.5a

and b). This did not occur in the axial areas, which were not subjected to masticatory stress (Fig. 6.5c).

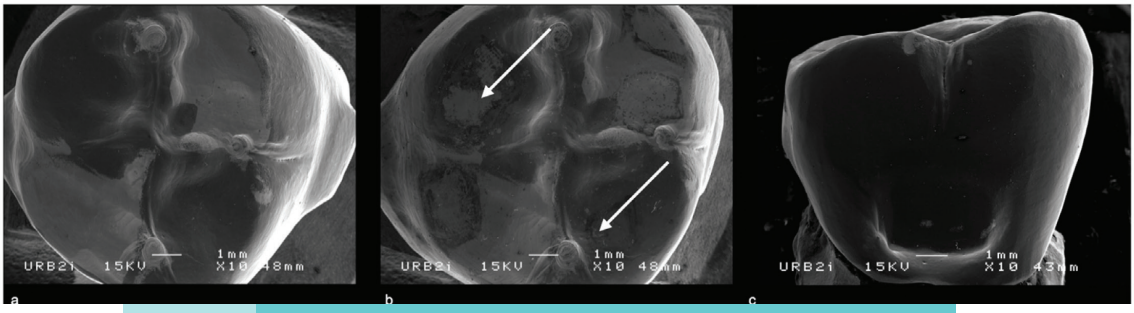


Fig 6.5 *SEM observation in a clinical case.*
(a) Occlusal areas at baseline. Light grey zones correspond to unglazed areas.
(b) Occlusal areas at 2-year follow-up: glaze is shown to wear out on occlusal contact points (white arrows).
(c) Buccal area at 2-year follow-up: glaze is shown to be intact.

3.3. Raman mapping

Images arising from Raman wide mapping (100 × 100 μm^2), high-resolution mapping (25 × 25 μm^2), and depth mapping of some detected transformed grains are presented in Fig. 6.6. The yellow and red images were combined to provide the spectral maps (Fig. 6.6c, e, and i). The peaks at approximately 180 cm^{-1} in the yellow spectrum were assigned to the monoclinic phase (Figs. 6.6e and 6.7i). The analyses revealed the presence of localized clusters of transformed grains (yellow spots) on both the axial and occlusal areas of the crowns subjected to 3 years of aging ; more clusters were observed in the axial area than in the occlusal area (Fig. 6.6f and j). No transformation was detected in the baseline sample (Fig. 6.6k). Depth mapping (up to 60 µm) revealed that the transformation propagated linearly from the surface to the interior (Fig. 6.6g and k).

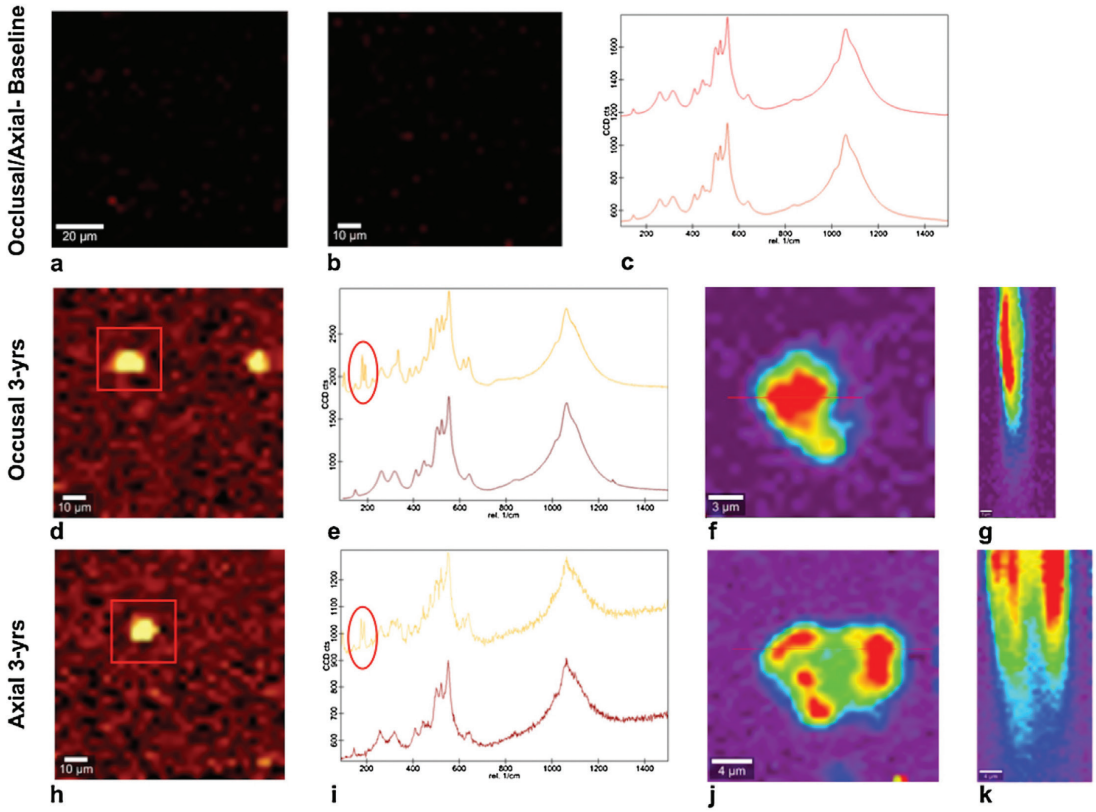


Fig 6.6 *Raman mapping on dental crowns at baseline versus at 3-year follow-up. Large-scale image (100 × 100 µm^2) with a scale from black (V_{fm}= 0%) to bright orange (V_{fm}= maximum):*
(a) Occlusal area at baseline;
(b) Axial area at baseline;
(d) Occlusal area at 3 years;
(h) Axial area at 3 years. Related spectra are presented in figures c, e, and f, respectively. The yellow spectrum shows peaks around 180 cm^{-1}, which are characteristic of the monoclinic phase (encircled in red). No t-m transformation was detected at baseline. High-resolution mapping (25 × 25 µm^2) was carried out on the detected transformed grains (encircled in red) with a scale from violet (V_{fm}= 0%) to red (V_{fm}= maximum) on the occlusal (f) and axial(j) areas, and depth mapping (60 µm) was performed along the red line (i and k, respectively).

3.4. High-resolution SEM observations without gold coating

High-resolution SEM images (Fig. 6.7) illustrate the effect of tribological damage on the occlusal contact areas, showing grain pull-out and crushed

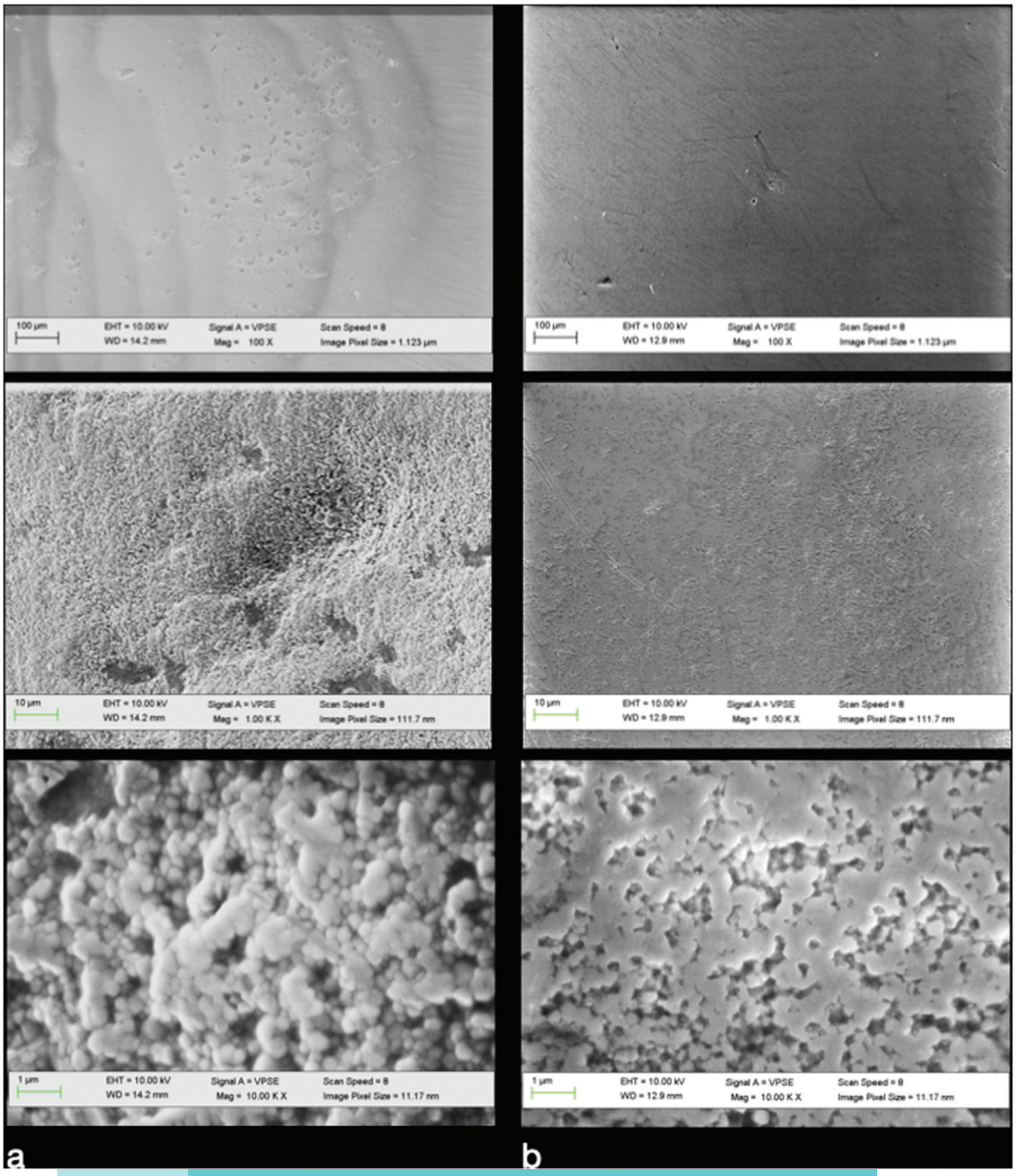


Fig 6.7 *High-resolution SEM observation of a dental crown at 3-year follow-up.*
(a) Axial area.
(b) Occlusal area. On the axial areas, which were not subjected to mechanical stress, the grain aspect is typical of the classical LTD process. However, on occlusal areas, SEM images illustrate the effect of the tribological stress on occlusal contact areas with grain pull-out, low-density material, and crushing of remaining grains, which appear plastically deformed.

grains due to plastic deformation. In the axial areas, which were not subjected to mechanical stress, there was less grain pull-out and no crushing.

4. DISCUSSION

4.1. Selection of phase transformation measurement method

X-ray diffraction (XRD) and Raman spectroscopy are the two most commonly used techniques for analyzing and quantifying the zirconia phase transformation [48]. The limitations of XRD include the penetration depth of the X-rays, the spatial resolution (typically only the first 10 microns below the surface are analyzed on a large surface of at least 50 × 50 µm) [6,49], and the accuracy for evaluating the onset of transformation (it is not precise for monoclinic content lower than 3%) [6,36]. Although much less frequently used than XRD, Raman spectroscopy is an efficient method for studying zirconia LTD [50] especially because the resolution is higher (1 μm^2), and it is possible to focus on the first in-surface micrometer or to explore up to 70 µm in depth with the combination of an adjustable confocal aperture and different objectives [46]. The method can detect very localized transformed spots on the surface of the prosthesis in the early stages of transformation, and it also presents the possibility of studying convex surfaces with a detection threshold of 5% [47]. The complex morphology (with convexities and concavities) of the prostheses represents a significant challenge for the detection of the *t-m* transformation, especially when XRD measurements require flat surfaces. Other non-destructive methods for the analysis of the *t-m* transformation include optical interferometry and atomic force microscopy ; however, these techniques do not allow direct quantification of the *m*-phase content [49].

4.2. Development of *t-m* transformation in Y-TZP dental prostheses

This study demonstrated that *t-m* transformation in Y-TZP dental prostheses developed over time ; the percentage of MPs+ (*n* tot = 2640 MPs) and the mean V_{fm} significantly increased with the duration between baseline and 2 years ($p < 0.0001$ and $p = 0.012$, respectively). The percentages of MP+ and MP+50% significantly increased between 6 months and 2 years ($p = 0.0001$ and $p = 0.0068$, respectively) (Table 6.5, Fig. 6.4). The bearing area of the curves between baseline and 6 months (Fig. 6.4) probably indicates a latency period during which the water diffuses into the material. At baseline, the *m* phase was significantly correlated ($p = 0.0003$) with the grinding procedures using a diamond bur to adjust occlusal contacts with antagonistic teeth, which generates stress-induced transformation and, importantly, microcracks [24]. The recommended zirconia regeneration firing procedure (1000 °C for 15 min when the prosthesis is sent back to the dental laboratory) [24] is debatable because it does not affect these microcracks. It must be noted that despite the use of the adjusted provisional restorations as a mock-up for the design of the zirconia restorations, 34% of occlusal areas had to be adjusted due to high occlusion, which is one of the limitations of CAD-CAM processes. The *m*-phase measurements obtained at 6 months can be compared with those reported in a study by Miragaya et al. [42], in which flat Y-TZP specimens were attached to personalized intra-oral resin appliances and exposed to the oral cavity (but not to mechanical stress) for two months. The material studied was the same (Lava Plus), and the authors also detected a *t-m* transformation using XRD (4.7% wt% at two months versus $V_{fm} = 0.1 \pm 0.9\%$ in the axial areas, i.e., not subjected to mechanical stress in the present study) (Table 6.4). The two-year results can be compared with those reported by Keuper et al. [51] in their *in vitro* study in which XRD was used to investigate the LTD of a second-generation Y-TZP dental ceramic (Vita YZ, Vita Zahnfabrik) after two years of storage in distilled water at 37 °C. They determined a V_{fm} of 28.1%, which is higher than the mean V_{fm} measured in areas not subjected to mechanical stress in the present study ($V_{fm} = 1.2 \pm 8.1\%$) (Table 6.4). This

difference can be explained by the different zirconia used and the higher measurement depth (5 or 7 µm) from the surface.

There was no significant difference between the percentages of transformation-positive MPs in the glazed and unglazed areas, globally, and when the axial and occlusal areas were considered separately, irrespective of the evaluation time. Consequently, glazing cannot be considered a protection against LTD. In occlusal areas, this can be explained by the fact that glaze rapidly wore out in most of the areas after 6 months (70.2% of cusps following SEM analyses) and in all of them after 1 year, as predicted by Denry and Kelly [24] (Fig. 6.5). Investigations regarding the influence of the finishing procedure showed that polishing should be preferred to glazing [52–58], which results in a higher surface roughness and antagonistic tooth wear [59,60]. However, in axial areas, glazing did not wear out, and the hypotheses for LTD-development in those areas are the diffusion of water through the thin glaze layer or the effect of glaze-layer firing, as suggested by some authors [61,62]. These results contradict those of an *in vitro* study [33], in which crowns subjected to mechanical stress showed a significant increase in the monoclinic phase in areas with glaze wear, in contrast to the specimens with intact glaze, which showed a stable level of the *m* phase.

4.3. Distribution and penetration of the *t-m* transformation

Regardless of the follow-up time, most of the transformation-positive areas presented several zero values for the five MPs of the *t-m* transformation, indicating a non-uniform process (Table 6.3). This explains the low mean V_{fm} values and the important standard deviations, which makes the mean V_{fm} values difficult to interpret (Table 6.4). Indeed, Raman mapping revealed the presence of localized clusters of transformed grains (yellow spots) on both the axial and occlusal areas of the crowns subjected to 3 years of aging (Fig. 6.6f and j). Clusters can result from the autocatalytic nature of the

nucleation and growth process of the phase transformation: transformation nucleation on a particular grain at the surface leads to a volume increase that stresses the neighboring grains, and this induces micro-cracking and further transformation [2]. These results confirm the *in vitro* observations of Zhang et al. [63] in which Raman mapping showed the heterogeneous development of *t-m* transformations after the samples were aged in the water at 90 °C for 7 days. Depth mapping (up to 60 µm) revealed that the transformation propagated from the surface to the interior (Fig. 6.6g and k), with the underlying tetragonal grains existing in tension and water penetrating through the micro-crack network. This promotes the development of LTD to the interior [2].

4.4. Influence of mechanical stress on the evolution of *t-m* transformation

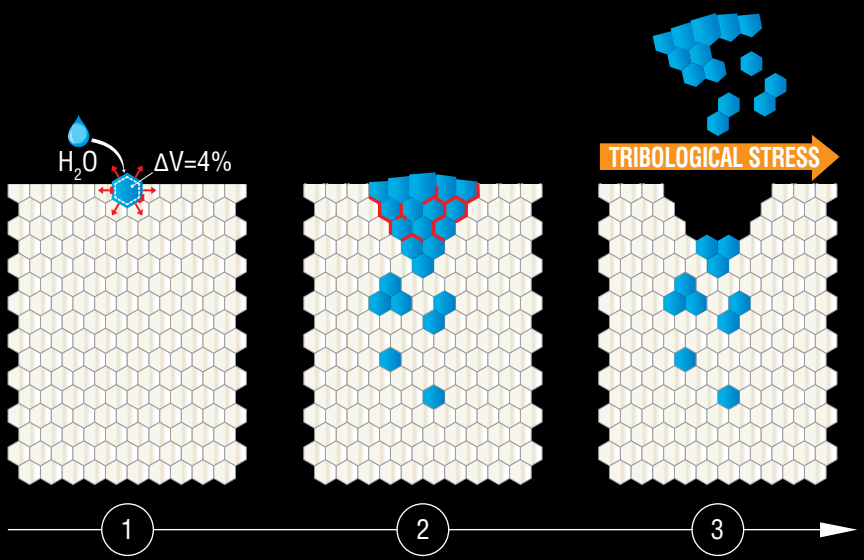
The transformation evolved differently in axial areas (not subjected to mechanical stress) than in occlusal areas (subjected to mechanical stress). The change in the percentage of MPs+ and the mean percentage of V_{fm} with time varied with the type of area studied (p = 0.0013 and p = 0.0016, respectively). In particular, in axial areas, the percentage of MPs+ was shown to significantly increase with time, while it was shown to remain stable in occlusal areas. However, both types of areas showed a significant increase between 6 months and 2 years (Table 6.5, Fig. 6.4a). The change in the percentage of MPs+50% with time increased in axial areas (statistical analysis not appropriate), whereas it was shown to significantly decrease between baseline and 6 months in occlusal areas (p = 0.034) and then to remain stable (Table 6.5, Fig. 6.4b). The mean V_{fm} remained stable in the axial areas and significantly decreased with time in the occlusal areas.

Consequently, it was postulated that the masticatory stresses generated grain pull-out from the clusters of transformed grains to explain those results. The high-resolution SEM images (Fig. 6.7), which were obtained to confirm this hypothesis, illustrate the effect of tribological damage on occlusal contact areas. They show a low-density material due to grain pull-out and

crushing of the remaining grains, which appear plastically deformed. Interestingly, the SEM images in occlusal areas are similar to observations in hip prostheses, which are also subjected to tribological stress [6,8,64,65]. Particularly, the images in Fig. 6.4b are comparable to FEG-SEM images of a fractured Y-TZP hip prosthesis 3 years after placement [65], which showed agglomerates of zirconia grains surrounded by a matrix of low-density and porous material, with the porous zone extending up to 0.5 mm beneath the original surface and being crossed by cracks. It is probable that the LTD initially induces surface roughness, micro-cracking, and, subsequently, tribological stress generates a pull-out of monoclinic grains as the transformation progresses (Fig. 6.8).

In the axial areas, which were not subjected to mechanical stress, the grain aspect was more typical of the classical nucleation-growth process of LTD, with the presence of porous material but less grain pull-out and no crushing

Fig 6.8 *Schematic illustration of the low-temperature degradation process in the presence of mechanical stress, which is characterized by a grain pull-out from clusters of transformed grains.*



as compared with occlusal areas. Raman mapping showed more clusters of transformed grains in the axial areas than in the occlusal areas.

In conclusion, one of the important findings of this study is that the absence of a detected monoclinic phase does not necessarily imply that LTD does not occur, and the presence of the *m* phase is not the sole parameter for detecting LTD.

4.5. Clinical impact of LTD

It seems that the development and progression of LTD in second-generation dental zirconia is quite low at the lifespan of dental prostheses, particularly if restoration thickness is important. In the present study, the main issue pointed out in the first publication related to the clinical results of this study was zirconia's high stiffness and inability to absorb occlusal stress, which was suspected to promote registered failures, such as implant loss, root fracture, core fracture, and antagonistic teeth failures [45]. It must be noticed that among those registered failures, one crown fracture and particularly one minor chipping [45] could have been promoted by LTD. However, fractures and LTD cannot be correlated in the present study, given that *t-m* measurement is not the sole parameter for detecting this process. Recently, Hansen et al. [66] studied 84 anterior monolithic zirconia crowns (BruxZir®, Glidewell Laboratories, USA) in heavy grinders with severe tooth wear, with a mean follow-up of 20 months. They observed 1.3% of total fractures (one crown fractured after 16 months) and 5.2% of chipping on roughened areas on the palatal face of the incisal edge, 1 month after placement in one patient. This chipping rate is high and surprising, particularly for high-strength materials such as zirconia. The authors also hypothesized that these changes could be due to the combined effect of occlusal stress and LTD on microcracks induced by the manufacturing process. If LTD was shown to affect dental prostheses in the same way as orthopedic prostheses, the clinical impact

of this phenomenon is unknown and needs to be evaluated through a thorough analysis of fractured prostheses (like the explant analyses performed with hip prostheses) in the framework of long-term and large-scale studies. Hopefully, from a biological point of view, and in the case of dental prostheses, zirconia debris are swallowed and cannot generate chronic inflammation and osteolysis like hip prostheses.

Finally, it must be emphasized that zirconia materials are sophisticated materials with varying compositions, microstructures, and manufacturing processes, which can significantly influence their properties and aging resistance. Therefore, the conclusions of the present study should not be extrapolated, particularly, to third-generation materials with more cubic phase (which are supposed to be less sensitive to LTD), and further investigations with different materials are required. The results obtained after 5 years in the present study will provide more information on long-term degradation.

5. CONCLUSION

Zirconia is a sophisticated and sensitive ceramic material, and the study of its low-temperature degradation (LTD), when used in biomedical prostheses, remains a challenge. Laboratory studies cannot be enough to predict clinical performance because *in vitro* aging conditions are far away from the body environment. In the present study, the oral cavity was shown to constitute an accessible and interesting experimental opportunity because, contrary to orthopedic prostheses, the restorations were easily and temporarily removed to perform *ex vivo* monitoring of LTD development. The results showed that LTD developed in monolithic 3Y-TZP restorations 6 months after intraoral placement and increased with time, while glazing did not protect from this process. After two years, the tetragonal-to-monoclinic transformation was

shown to be non-uniform, with the presence of localized clusters of transformed grains. However, there was a significant difference between the LTD process in the occlusal areas and the areas free of masticatory stress. In the axial areas, the grain aspect was typical of the classical nucleation-growth process reported for LTD, which evolved from the surface to a depth of several tens of microns. However, in occlusal areas, tribological stress generated surface crushing and grain pull-out from the clusters, as observed in failed hip prostheses, and this may allow the underestimation of the risk of LTD when evaluated by *m*-phase quantification with XRD or Raman spectroscopy. Results also highlight that *m*-phase quantification is not the sole parameter indicative of LTD. If LTD occurs in dental prostheses in the same way as in orthopedic prostheses, its clinical impact is unknown and needs to be evaluated through a thorough analysis of fractured prostheses in the framework of long-term studies.

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Clinical behavior of second-generation zirconia monolithic posterior restorations: Two-year results of a prospective study with *ex vivo* analyses including patients with clinical signs of bruxism

Vinciane Koenig [1, 2, a]
Claudine P. Wulfman [1, 3, a]
Sandrine Bekaert [1, 2]
Nathalie M. Dupont [1, 2]
Stéphane O. Le Goff [3]
Maher Eldafrawy [1]
Alain J. Vanheusden [1, 2]
Amélie K. Mainjot [1, 2]

[1] *Dental Biomaterials Research Unit (d-BRU), University of Liège (ULiège), 45 Quai G. Kurth, Liège, 4020, Belgium*

[2] *Department of Fixed Prosthodontics, Institute of Dentistry, University of Liège Hospital (CHU), 45 Quai G. Kurth, Liège, 4020, Belgium*

[3] *Unité de Recherches en Biomatériaux Innovants et Interfaces (URB2i) – EA4462, Faculté de Chirurgie Dentaire, Université Paris Descartes, Sorbonne Paris-Cité, Montrouge, 92120, France*

a These authors contributed equally.

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ABSTRACT

Objectives: This study aimed to investigate (1) clinical outcomes of second-generation zirconia restorations, including patients with bruxism clinical signs, and (2) the material wear process.

Methods: A total of 95 posterior monolithic zirconia tooth-elements in 45 patients were evaluated, 85 on implants and 10 on natural teeth, and 20.3% of restorations being fixed partial dentures (FPDs). Occlusal contact point areas were determined and half of those areas were left unglazed and just polished. Restorations were clinically evaluated following criteria of the World Dental Federation and antagonistic teeth were examined at each evaluation time. Wear *ex vivo* analyses using SEM and 3D laser profilometry were performed at baseline and after 6 months, 1 year, and 2 years respectively, temporarily removing the prostheses.

Results: The Kaplan-Meier survival rate of restorations was 93.3% (100% for FPDs) and the success rate was 81.8%, with 4 abutment debondings, 3 tooth-supported crown debondings (provisional cement use), 1 restoration fracture, 1 minor chipping, 1 core fracture, 1 root fracture, and 2 implant losses. 80% of catastrophic failures occurred in patients with clinical signs of bruxism (61.7% of patients). Complications were also observed on antagonistic teeth (3 catastrophic failures). Clinical evaluation of the restorations showed good results from the aesthetic, functional, and biological perspective. Zirconia wear was inferior to 15 µm, while glaze wear was observed on all occlusal contact areas after 1 year.

Conclusions: Monolithic zirconia FPDs are promising but the succes rate of single-unit restorations was not as high as expected in this sample including patients with bruxism clinical signs.

Clinical significance: Within study limitations, FPDs showed excellent short-term results but further research is needed for single-unit restorations considering samples, which do not exclude bruxers. The weak link is the restoration support or the antagonist tooth, one hypothesis being that zirconia stiffness and lack of resilience do not promote occlusal stress damping.

Keywords: Dental prosthesis, Zirconia, Computer-Aided design/Computer-Aided manufacturing Wear

1. INTRODUCTION

Three mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) was introduced in prosthodontics in the early 2000s as an alternative to metal due to its high strength and biocompatibility combined with a white appearance. 3Y-TZP has original properties since it is in a metastable state at room temperature and can transform from a tetragonal to a monoclinic (*t-m*) crystalline form under the effect of stress, giving zirconia high toughness in comparison with other ceramic materials. However, this transformation can also occur in a very slow process when the material is in contact with water: this aging phenomenon, called low-temperature degradation (LTD), can engender material surface alterations and fracture [1]. The first clinical reports about veneered zirconia-based crowns and fixed partial dentures have indicated a high rate of short-term failures due to a high rate of cohesive fracture of the veneering ceramic (chipping), which constitutes the weak link of the restoration [2–6]. This problem was confirmed with long-term studies [7–9].

Even if some recommendations have been formulated to reduce this problem, monolithic zirconia restorations have recently been introduced to avoid chipping and reduce fabrication costs [10]. However, this required the development of new zirconia materials with high-translucency properties. At present, there is a wide range of zirconia materials on the market in terms of composition and microstructure, and consequently properties.

Currently, three generations of zirconia materials are described [11]. First-generation zirconia is the original (3Y-TZP), which is used to produce frameworks for veneered restorations. The main characteristics of this generation are the low content in the cubic phase (< 15%), the resulting high strength (1200 MPa) and toughness and a high opacity and refractive index. To improve translucency, the alumina content was reduced and/or the sintering temperature was increased to reduce porosities while increasing

the grain size [12], introducing the second generation [11]. The aesthetic properties improvement allows for the fabrication of posterior monolithic restorations but changes in material composition and microstructure challenge resistance to LTD due to a higher metastability [12–14]. Aesthetic anterior monolithic restorations are now accessible using third-generation zirconia, which is even more translucent while resistant to LTD due to a higher cubic phase content (partially stabilized zirconia 4 mol% or 5 mol%: 4-YPSZ contains more than 25% of the cubic phase and 5-YPSZ more than 50%). However, the resulting mechanical properties are lower than the two previous generations (< 700 MPa flexural strength for 5-YPSZ), since the cubic phase does not undergo stress-induced transformation.

Most publications reporting clinical outcomes with zirconia monolithic prostheses described restorations made from second-generation zirconia [15–28]. However, patients presenting high occlusal stress, such as those with bruxism, were included in only one study [28], which engenders important bias. Indeed, bruxism is reported to have a high prevalence [29], while manufacturers often recommend monolithic zirconia restorations for this indication.

Finally, material and antagonistic tooth wear was also a concern when zirconia monolithic restorations were introduced. Publications reporting *ex vivo* quantitative measurements of wear showed negligible wear of zirconia surfaces [20] and less abrasive effects on antagonist teeth than other ceramic materials; both polishing and glazing as finishing procedures were investigated [20,24,27,30]. Nevertheless, those measurements were performed on replicas, which was shown to engender significant imprecision in wear evaluation and counteracted SEM observations [31].

Consequently, an original prospective clinical study protocol that includes *ex vivo* analyses of the restoration was designed [32] to investigate (1) in-mouth LTD process of second-generation zirconia restorations on teeth and

implants (2) biological, functional, and aesthetic clinical outcomes in patients with and without clinical signs of bruxism and (3) material wear. The present work reports the 2-year results related to clinical outcomes and wear analysis.

2. MATERIALS AND METHODS

2.1. Study design

The protocol of this prospective study was approved by the Ethics Committee of the University Hospital Center (CHU) of Liege and was registered on the ClinicalTrials.gov database (Identifier NCT02150226). The complete study design and protocol were previously published in detail [32]. The determination of the sample size was based on the LTD outcome. Written patient consent was obtained before inclusion and 47 patients were included from February 2014 to December 2015. The patients were treated by four experienced operators from the Department of Fixed Prosthodontics, Institute of Dentistry, CHU of Liege, Belgium. The eligibility criteria were the need for molar or premolar crowns (maximum 6 elements per patient). Restorations were realized either on teeth or implants and multi-unit restorations were also included, provided they were on implants and were limited to 3 elements (maximum 2 fixed partial dentures (FPDs) per patient). The exclusion criteria were severe or acute periodontal or carious diseases and poor oral hygiene. Patients with removable prosthesis as an antagonist were excluded. The participants received no financial compensation, although treatment and prostheses were free of charge.

2.2. Occlusal risk factors

The presence of bruxism was evaluated through clinical examination and self-reporting [33]. The presence of bruxism was recorded if the patient fulfilled at least two criteria: (1) reporting teeth grinding during the night or day and (2) the presence of at least one clinical sign among the following: abnormal attrition wearing facets on teeth, transitory pain or fatigue on waking felt in the jaw muscles, temporal headaches on waking, or jaw locking on waking related to teeth grinding during sleep [34].

Occlusal relationships were characterized as favorable or unfavorable based on the clinical examination. Class III or class II.2 malocclusion, anterior or posterior crossbite, edge to edge or open bite were considered as unfavorable occlusal relationships. The use of an occlusal nightguard was noted.

2.3. Clinical procedure and fabrication of the restorations

All of the clinical and technical procedures were performed in strict agreement with the clinical and technical instruction protocol validated by the ethics committee and following the manufacturers' recommendations. Teeth were prepared following standardized criteria (1.0–1.5 mm occlusal depth cut to achieve the appropriate occlusal anatomy, 1.0–1.5 mm functional cusp tip reduction, 0.5 mm gingival chamfer reduction, and a 6–8 degree taper to the axial walls). A double-mix impression was performed with high- and a low-viscous A-silicone impression material (Aquasil Heavy/XLV, Dentsply De Trey, Konstanz, Germany) and the same impression procedure was used for implant restorations. Two implant systems (NobelReplace, Nobel Biocare, Gothenburg, Sweden; and Standard Implants, Straumann, Basel, Switzerland) were used in this clinical study. Shade was registered with the Vita Classic System (Vita Zahnfabrik, Bad Säckingen, Germany). Restorations on antagonistic teeth were replaced in the presence of a deficient marginal joint, decay, or unadapted morphology.

CAD-CAM composite provisional crowns (Lava Ultimate, 3M, Seefeld, Germany) or PMMA provisional FPDs were designed with specific buccal and palatal grips to facilitate cemented crown removal [32]. Provisional restorations were in-mouth adapted, particularly regarding occlusal adjustments, and used as a model for the design of the zirconia restorations (Lava Plus, 3M, Seefeld, Germany). Lava Plus High Translucency Zirconia is a tetragonal polycrystalline zirconia partially stabilized with 3 mol% Yttria engineered for high translucency and utmost strength. It has a lower Alumina content of 0.1% compared to Lava Frame Zirconia, optimally distributed within the material for maintaining aging stability. Sintering was performed following the manufacturer's instructions at 1450 °C for 2 h. The zirconia restorations were tried in and if needed occlusal contact points were adjusted and polished. Firing of the glaze (IPS Empress stains and e.max Ceram Glaze, Ivoclar Vivadent, Schaan, Liechtenstein) at 780 °C for 1 min completed the manufacturing process. Bonding to titanium abutments (1000er-Serie, Medentika, Hugelsheim, Germany) was performed with a resin composite cement, either RelyX Ultimate (3 M, Seefeld, Germany) in the first part of the study, or Multilink Hybrid Abutment (Ivoclar Vivadent, Schaan, Liechtenstein) in the second part, following the manufacturers' recommendations, that is, after sandblasting the abutment and the zirconia restoration with 50 µm alumina particles. The occlusal surface contact areas, which were not glazed, were randomly determined (Fig. 7.1a). Four occlusal contact points (one contact per cusp) were determined on the molars and two on the premolars (Fig. 7.1b). For molars, two cusps were randomly selected to remain unglazed: one centric cusp (unglazed centric cusp, UCC) and one non-centric (unglazed non-centric cusp, UNCC). The two other cusps were called the glazed centric cusp (GCC) and glazed non-centric cusp (GNCC). For premolars, one cusp was randomly selected to remain unglazed. Control areas were the buccal face (glazed) and the lingual/palatal face (unglazed) of the restoration.

Baseline analyses were performed before placement (see Section 2.4). Cemented restorations were sealed with eugenol-free cement (RelyX Temp

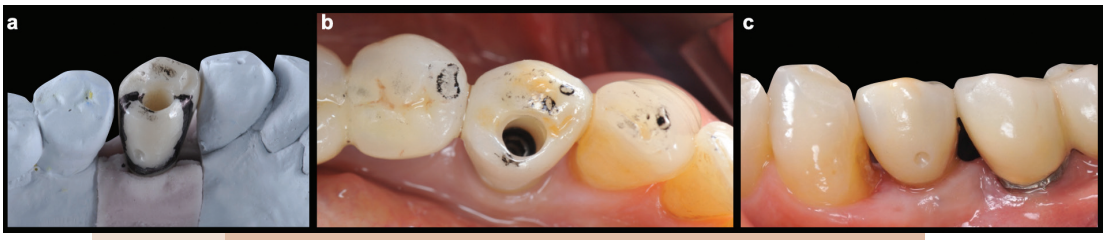

Fig 7.1 *Screw-retained crown on implant (tooth #34).*
a) Landmarking with permanent ink of areas, which will not be glazed.
b) Occlusal contact points.
c) Aesthetic integration. Photo courtesy of A. Mainjot.

NE, 3 M), while screw-retained restorations were torqued at 35 Ncm^{-1}. Clinical evaluation was performed one week after placement (see Section 2.3). After 6 months, the restorations were clinically evaluated and then removed for *ex vivo* analyses. Provisional restorations replaced zirconia restorations during *ex vivo* analyses. After these analyses, zirconia restorations were placed again in the mouth of the patient. The same procedure was repeated after 1 and 2 years. All of the data were recorded in a specifically designed online database, so that clinical data, pictures, and *ex vivo* measurements could be easily available for consultation. The database is hosted on the university hospital secured server.

2.4. Clinical outcomes

2.4.1. Restoration evaluation

Clinical evaluation followed World Dental Federation recommendations and used World Dental Federation Instruments for assessing dental restorations [35]. This instrument is comprised of three dimensions (18 items): biological (six items), functional (seven items), and aesthetic (five items). The two items related to wear were not considered since the quantification was performed *ex vivo*, so only 16 items were reported in the study. Each item was assessed

by clinical examination on a 5-point Likert scale (1 corresponding to a perfect restoration and 5 corresponding to a restoration that needs to be replaced). Two independent evaluators performed the evaluations after training on the e-calib web-based software and group training sessions. The type of support (tooth or implant) was registered. In addition to radiographs, pictures were taken of the restorations and the antagonistic teeth with the occlusal contact point registering.

2.4.2. Antagonistic teeth evaluation

The nature of the antagonistic teeth was registered at baseline. All of the complications and interventions were recorded.

2.4.3. Patient satisfaction level

The FDI evaluation included patient self-reported satisfaction, on a 5-point Likert scale, regarding the aesthetic appearance of the crown and/or function. Therefore, the criterion was divided into two subscores [35].

2.5. *Ex vivo* analyses

2.5.1. SEM observations

The removed crowns were observed with an JSM-6400 scanning electron microscope (JEOL Limited, Tokyo, Japan) to monitor the wear and glaze coating evolution.

2.5.2. Profilometry

For zirconia wear quantification, the restorations were scanned *ex vivo* with a custom-made device including an XY motorized board stage and a

100 nm resolution laser sensor (Keyence LK G30 with LK GD500 controller, Keyence Corporation, Osaka, Japan) with a step of 25 µm. The restorations were placed in a specific holder to ensure positioning reproducibility at each evaluation time. Raw data acquisition and processing were performed using a custom-developed software with C# language (Microsoft Visual Studio 2013, Microsoft Corporation, Redmond, WA, USA) coupled to a digital data-acquisition PCI board (NI PCI-6534, National Instruments Corporation, Austin, TX, USA). The resulting matrix of Z values was then transferred to the surface-matching software Geomagic Control 2015 (Geomagic Inc, Morrisville, NC, USA). The same operator (CW) performed all of the wear quantifications. Baseline scans were transformed into a computer-aided design format (STL) and recall scans were superimposed using a best-fit alignment algorithm. First, the software randomly selected and aligned 300 data points. After this rough alignment, fine alignment using 1000 additional data points was performed by iterative rotations and translations, minimizing the root-mean-squared difference between the two images. The deviation eliminator function was used to choose data points with a minimum deviation in the Z-axis. The matching process was considered acceptable if the root mean square was inferior to 20 µm. Only scans that successfully passed this matching process step were used for the wear analysis.

To prevent bias in the wear measurement related to artifacts or surface pollution, a threshold value of 150 µm was defined, leading to the exclusion of points with a measured difference superior to 150 µm from the wear evaluation. An additional control for adequate matching was the distribution of the z-values in the areas that were not subjected to wear (for example, occlusal grooves). In these areas, data points with a difference in z-values superior to 15 µm were excluded. A mean wear value was registered both for the entire occlusal surface and for each occlusal contact area (OCA), which were identified from the clinical pictures. These areas were digitally designed on each baseline scan using a mask.

2.5.2.1. Accuracy and precision calibration

The accuracy and precision of the method were assessed in a series of three experiments using the reference-free superimposition algorithm of Geomagic software according to the protocol proposed by Rosin et al. [36]: (1) precision

Sample description	% (n)
Patients (n = 47)	
Sex	
Female	70.2 (33)
Male	29.8 (14)
Bruxism (self-report + clinical inspection)	61.7 (29)
Nightguard	19.1 (9)
Restorations (n = 75)	
Crowns	
Implant screw-retained crowns	58.7 (44)
Implant cemented crowns	5.3 (4)
Cemented crowns on teeth	17,3 (13)
Bridges	
Screw-retained bridges on two implants	17.3 (13)
3-element	14.7 (11)
2-element	2.7 (2)
3-element cemented bridge on two implants	1.4 (1)
Tooth elements (n = 101)	
Support	
Tooth	12.9 (13)
Implant	87.1 (88)
Tooth type	
Premolar	38.6 (39)
Molar	61.4 (62)
***Ex-vivo* analyzed occlusal areas (n = 326)**	
GCC	26.4 (86)
UCC	23.9 (78)
GNCC	29.1 (95)
UNCC	20.6 (67)
Antagonists (n = 127)	
Tooth	88.2 (112)
Implant	11.8 (15)

Table 7.1 *Sample description in terms of patients, restorations, tooth-elements, antagonists and ex vivo analyzed occlusal areas. GCC: glazed centric cusps, UCC: unglazed centric cusps, GNCC: glazed non-centric cusps, UNCC: unglazed non- centric cusps.*

of the automatic 3D superimposition algorithm, (2) precision of the 3D data acquisition, and (3) precision of the reference-free 3D superimposition.

2.6. Statistical analysis

The results are presented as means and standard deviations (SD) for the continuous variables and as frequency tables for the categorical variables. Comparisons between two categorical variables were done using Fisher's exact test. Statistical analysis differences between the experimental groups were assessed using InStat software (GraphPad), SAS version 9.4. An additional Kaplan-Meier analysis of the survival and success rates was performed.

3. RESULTS

3.1. Clinical data on patients and restorations

A total of 47 patients were recruited (14 male and 33 female), with a mean age of 54.34 years (SD 15.32). Overall, 75 restorations corresponding to 101 elements were included during the inclusion period from February 2014 to December 2015. Sample descriptions in terms of patients, restorations, tooth elements, antagonists, and *ex vivo* analyzed occlusal areas are presented in Table 7.1. At 2 years, 45 patients and 95 elements were evaluated. After 2 years, 1 patient dropped out of the study.

The patients presenting bruxism clinical signs represented 61.7% of the cohort. All presented abnormal attrition wear facets. Among those patients, 19.1% had a nightguard but only one regularly wore it. Finally, 34% of patients presented unfavorable occlusal relationships.

3.2. Clinical outcomes

3.2.1. Restoration evaluation

The restorations (n = 75) had a Kaplan-Meier survival rate (which comprises all restorations still in the mouth even if failed) of 93.3% ± 2.9% (5 restorations were lost) (Fig. 7.2a) and a Kaplan-Meier success rate (which takes into account all failed restorations) of 81.8% ± 4.7% (Fig. 7.3a). 80% of the catastrophic failures and 76.9% of all of the complications occurred in patients with clinical signs of bruxism, while 20% of the catastrophic failures and 23.1% of all of the complications occurred in patients with unfavorable occlusal relationships. Those complications are presented in Table 7.2. The debonding

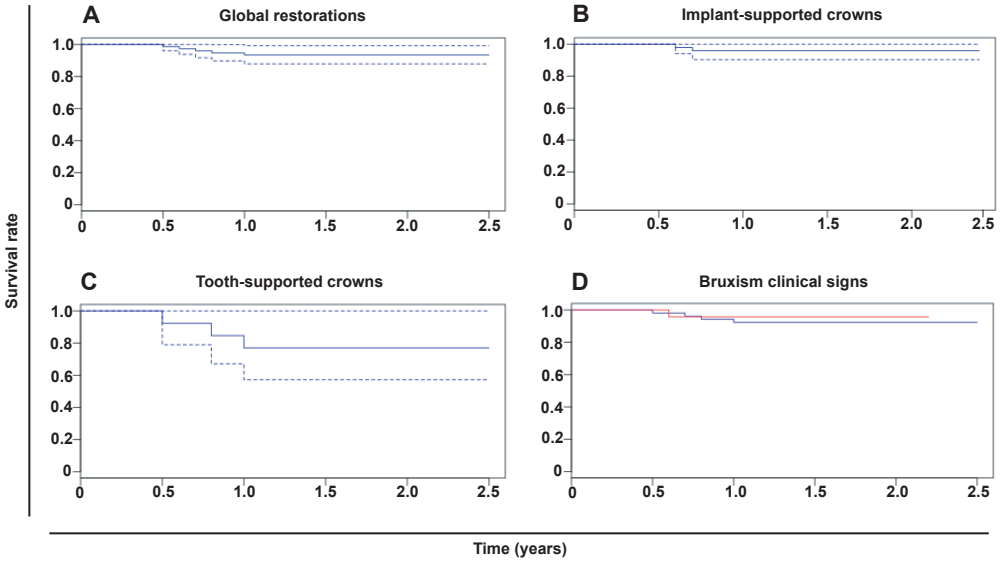

Fig 7.2 *Kaplan-Meier survival rate.*
a) Global.
b) Implant-supported crowns.
c) Tooth-supported crowns.
d) Comparison between restorations inserted in patients with (in blue) or without parafunctional habits (in red). Dotted lines represent 95% confidence intervals. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

issue, which is at the origin of most failures, was significantly related to the type resin composite cement used for titanium base bonding (p = 0.0046) and was solved replacing RelyX Ultimate by Multilink Hybrid Abutment (Ivoclar Vivadent AG, Schaan, Liechtenstein). If not considering the tooth-supported crown debonding since crowns on the natural teeth were provisionally cemented for the study purpose, the success rate was 86.7%.

Complications	**% (n)**
Restorations	
Screw-retained restoration debonding from titanium base	5.3 (4)
Cemented restoration debonding	4.0 (3)
Implant loss	2.7 (2)
Crown fracture	1.3 (1)
Root fracture	1.3 (1)
Composite core fracture	1.3 (1)
Minor chipping	1.3 (1)
Antagonists	
Root fracture	1.6 (2)
Severe periodontal disease	0.8 (1)
Minor composite chipping	1.6 (2)

Table 7.2 *Percentage of complications.*

Restorations placed in the patients with bruxism clinical signs had a Kaplan-Meier survival rate of 92.3% ± 3.7% and a Kaplan-Meier success rate of 79.6% ± 5.9%, while those placed in the patients without bruxism clinical signs had a Kaplan-Meier survival rate of 95.7% ± 4.2% (Fig. 7.2d) and a Kaplan-Meier success rate of 87.0% ± 7.0% (Fig. 7.3d). However, this difference was not statistically significant regarding the survival rate (p = 0.60) or the success rate (p = 0.55)

Considering the results by the type of restoration, implant-supported FPD's had a survival rate of 100%, implant-supported crowns had a Kaplan-Meier survival rate of 95.8% ± 3.0% (Fig. 7.2b), and tooth-supported crowns had a Kaplan-Meier survival rate of 76.9% ± 12.0% (Fig. 7.2c).

Kaplan-Meier success rate for implant-supported FPD's was 100%, 84.3% ± 5.6% for implant-supported crowns (Fig. 7.3b) and 53.8% ± 13.8% for tooth-supported crowns (Fig. 7.3c), or 76.9% if debonding of provisionally cemented crowns were not considered.

The FDI scores of the restorations are presented in Table 7.3. Regarding the aesthetic properties, zirconia restorations showed excellent or good results (Fig. 7.1c) except for color match and translucency. Indeed, 65.2% were judged too bright but acceptable by the dentist. Regarding the functional

	Clinically Excellent % (n)	**Clinically Good % (n)**
A. Esthetic properties		
Surface luster	94,7 (90)	5,3 (5)
Staining		
a. surface	96,8 (92)	3,2 (3)
b. margin	96,8 (92)	3,2 (3)
Color match and translucency	5,3 (5)	295 (28)
Esthetic anatomical form	44,2 (42)	474 (45)
B. Functional properties		
Fracture of material and retention	99 (94)	
Marginal adaptation	95,8 (91)	4,2 (4)
Approximal anatomical form		
a. contact point	652 (62)	3,2 (3)
b. contour	99 (94)	1 (1)
Radiographic examination	100 (95)	
Patient's view	60 (57)	326 (31)
C. Biological properties		
Postoperative sensitivity and tooth vitality	90 (9)	
Recurrence of caries, erosion, abfraction	100 (10)	
Tooth integrity	100 (10)	
Periodontal response	98 (93)	1 (1)
Adjacent mucosa	99 (94)	
Oral and general health	100 (95)	

Table 7.3 *FDI scores of the aesthetic, functional, and biological properties after 2 years, in evaluated in 10 teeth.*

properties, zirconia monolithic restorations showed excellent or good results, except for the proximal contact points that were evaluated as too weak for 4.2% of the restorations. The biological properties demonstrated excellent results.

3.2.2. Antagonistic tooth evaluation

Three antagonistic teeth were lost due to root fracture (1.6%) or severe periodontal disease (0.8%) (Table 7.2). One was a monolithic zirconia restoration (Fig. 7.4h). These teeth were weakened by post and core restoration

	Clinically Sufficient % (n)	Clinically Unsatisfactory % (n)	Clinically poor % (n)	Acceptable %	Unacceptable %
				100	
	652 (62)				
	8,4 (8)				
				95,8	**4,2**
	1 (1)				
	274 (26)	4,2 (4)			
	7,4 (7)				
				89	**11**
		10 (1)			
			1 (1)		
	1 (1)				

percentage (n = 95). Postoperative sensitivity, recurrence of caries, and tooth integrity were

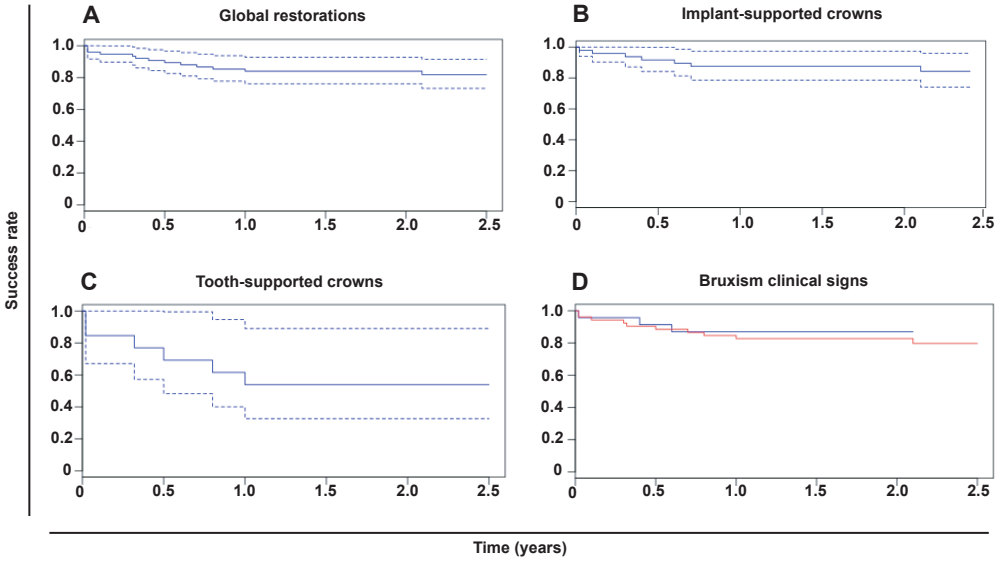

Fig 7.3 *Kaplan-Meier success rate.*
a) Global.
b) Implant-supported crowns.
c) Tooth-supported crowns.
d) Comparison between restorations inserted in patients with (in red) or without parafunctional habits (in blue). Dotted lines represent 95% confidence intervals. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

or already had periodontal disease before treatment. Two minor composite chippings were detected and repaired (1.6%) (Fig. 7.5a). Monolithic zirconia restorations that were located in the front of those teeth did not show any failures.

3.2.3. Patient satisfaction level

The FDI scores of the patients' views showed that 60.0% rated their satisfaction level as excellent, while 100% of the restorations were rated as acceptable. Only 2.1% reported minor esthetic issues because they judged their crowns as too bright.

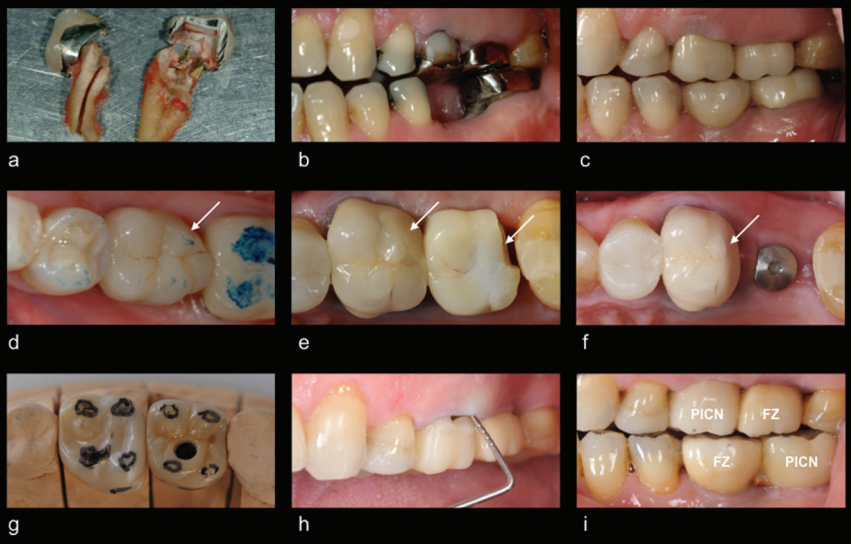

Fig 7.4 *Patient.*
(a and b) Root fracture #36 and extraction.
(c) Realization of veneered zirconia crowns on teeth #26, #27, and #37 and implant #36.
(d and e) Chipping (arrowed) on #26 and #36 after 6 months and on #27 after 3 years.
(f) Root fracture #27 after 8 years.
(g) Monolithic zirconia crowns #26, #27, and #36.
(h) Root fracture on tooth #26 after 1 year.
(i) Use of polymer-infiltrated ceramic network (PICN) material for crowns on implant #26 and implant #37 at a 2-year follow-up. Full zirconia (FZ) restorations on implant #27 and implant #36 at a 3-year follow-up. Photo courtesy of A. Mainjot.

3.3. *Ex vivo* analyses

Two screw-retained crowns were not *ex vivo* analyzed because their removal was not possible from a technical point of view.

3.3.1. SEM observations

After 6 months, 70.2% of the glazed cusps presented glaze wear with variable extents on the contact point areas. Glaze wear was observed in 100% of the glazed occlusal contact points after 1 year (Fig. 7.6).

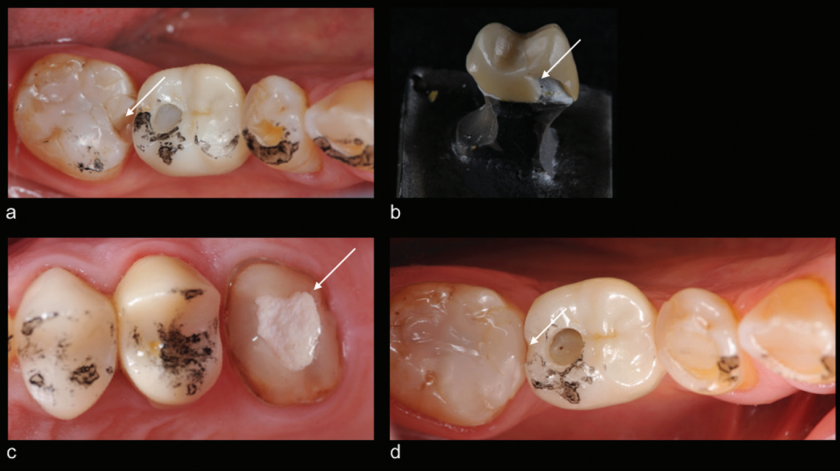

Fig 7.5 *Failures.*
a) Composite chipping on an antagonistic tooth (tooth #47).
b) Monolithic zirconia crown fracture (tooth #14), with a framework, which was not particularly thin.
c) Core debonding (tooth #16).
d) Minor chipping (tooth #46).
Photo courtesy of V. Koenig.

3.3.2. Profilometry

3.3.2.1. Accuracy and precision calibration.

For 3-dimensional measurements of zirconia wear, the intrinsic errors of the superimposition program of the software (experiment 1) resulted in an accuracy of 0.01 ± 0.01 µm (the accuracy is reported as the mean of the multiple measures and the precision corresponds to the standard deviation [37]). Assessment of the three-dimensional data acquisition produced differences in height of 0.09 ± 0.09 µm (experiment 2). The superimposition when the position of the crown within the laser scanner was altered after each scanning procedure (experiment 3) resulted in an accuracy of 0.47 ± 0.17 µm. The vertical resolution of the laser scanner was 15 µm. After 2 years, the wear of the zirconia prostheses occlusal face was inferior to the accuracy threshold of the measurement chain (15 µm). However, wear

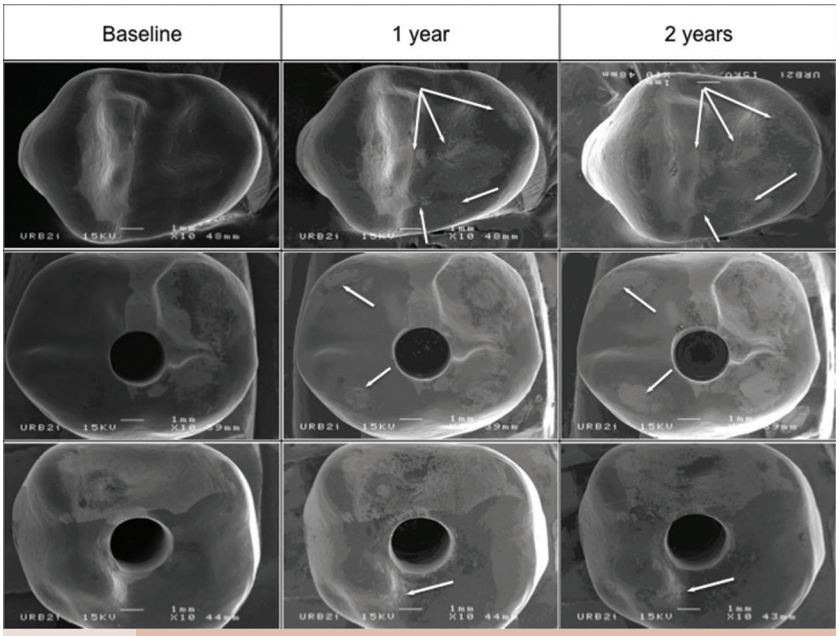

Fig 7.6 *Glaze wear. Glazed areas are darker on baseline SEM images (baseline). Arrows indicate wear zones after 1 and 2 years.*

was detectable on the occlusal contact points specifically for 15% of the investigated teeth (7 teeth) (Fig. 7.7). Finally, 16% of the scans were unusable for wear quantification due to their insufficient quality.

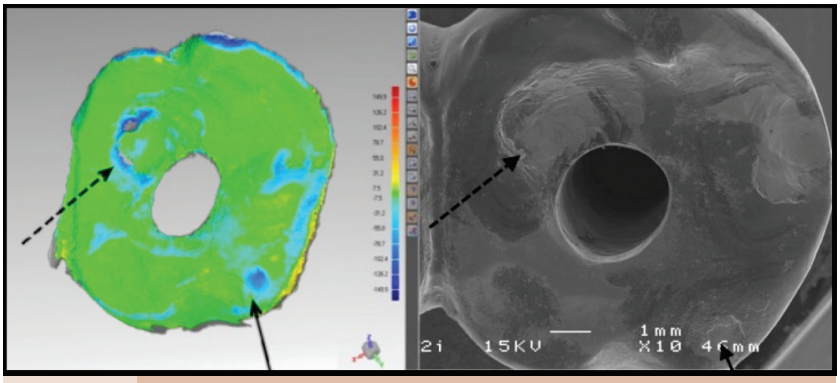

Fig 7.7 *Glaze wear. Glazed areas are darker on baseline SEM images (baseline). Arrows indicate wear zones after 1 and 2 years.*

4. DISCUSSION

Second-generation zirconia is widely used for posterior monolithic restorations but currently the clinical background is lacking and only one study included patients with bruxism, while manufacturers often recommend full zirconia restorations for this indication [15–28].

This study included 101 restorations in 47 patients followed for 2 years in a sample including 61.7% of patients with clinical signs of bruxism registered with a standard non-instrumental approach of bruxism diagnosis, including a clinical inspection and self-report, notably following the criteria of the American Academy of Sleep Medicine [34,38,39]. However, bruxism diagnosis is complex and inaccurate, and if the employed criteria are graded 2 on 3 in terms of assessment validity (meaning "probable sleep/awake bruxism"), polysomnography is recommended to confirm diagnosis [33], which was not possible in this large-scale clinical study. All of the patients recorded as bruxers presented abnormal attrition wear facets and reported teeth grinding during the night or day, which means that the presence of bruxism is probable but can engender some false positives [38]. Consequently, it can explain the high proportion of patients considered bruxers compared to the literature data, but those data are restricted, particularly for awake bruxism, and diagnostic criteria are greatly heterogeneous (reported prevalence is 22-30% for awake bruxism and 1–15% for sleep bruxism) [40].

Restorations (n = 75) had a global survival rate of 93.3% ± 2.9% (Fig. 7.2a) and a global success rate of 81.8% ± 4.7% (Fig. 7.3a), while patients with clinical signs of bruxism exhibited a lower restoration survival (92.3% versus 95.7%) (Fig. 7.2d) and success (79.6% versus 87.0%) (Fig. 7.3d) rates. If this difference was not significant (p = 0.60 and p = 0.55 for the survival and the success rate, respectively), long-term results are needed to study the influence of this parameter on restoration performance.

Implant-supported FPDs (*n* = 14) showed excellent results with a survival rate of 100%. This can be explained by the fact that splinting implants improve stress distribution [41]. Cardelli et al. presented encouraging results after 1 year in a pilot study of 2 patients rehabilitated with monolithic zirconia full-arch fixed prostheses on implants [17], as did Rojas Vizcaya in a 2- to 7-year retrospective study about 20 double full-arch prostheses [22]. The observed survival rate was better than the reported estimated 5-year survival rate of veneered zirconia and metal-ceramic implant FPDs (93.0% and 98.7%, respectively) [42], knowing that 4.1% of the zirconia implant-supported FPDs were lost due to ceramic or framework fractures.

Implant-supported crowns (*n* = 48) showed a survival rate of 95.8% ± 3.0% (Fig. 7.2b), which is inferior to the 100% 3-yr survival rate reported in the recent study of De Angelis et al. about 19 screw-retained restorations, which did not include patients with bruxism and constitutes, to author's knowledge, the only published study about monolithic zirconia implant crowns [18]. Loss of osseointegration was the only cause of failure, engendering 4.2% of implant loss. Implant loss was characterized by implant mobility as well as bone cratering visible on the radiograph. One implant was lost in a patient with bruxism signs who had a history of periodontal disease and peri-implantitis, and the other implant was a narrow implant that presented a beginning of bone loss after implantation. The implant-supported crown survival rate in this study was globally lower than the reported estimated 5-year survival rate of veneered zirconia and metal-ceramic implant crowns described in a recent meta-analysis (97.6% and 98.3%, respectively) [43]. In this review, the authors underlined the prevalence of veneering ceramic fractures for the zirconia crowns (2.8%) and noticed that 3.3% of the implants supporting metal-ceramic crowns and 4.3% of the implants supporting zirconia-based crowns experienced significant bone loss, defined as marginal bone levels more the 2 mm below what can be expected as normal bone remodeling. One hypothesis to explain the lower results of the present study is that in cases of high occlusal stress, the veneering ceramic constitutes the weak link of the

restoration and can act as a breaker, while in cases of monolithic restoration, zirconia being stiff, the stress is transmitted to the implant, engendering a higher rate of implant failures.

Tooth-supported crowns (n = 13) exhibited the worst survival rate: 76.9% ± 12.0 (Fig. 7.2c). Failures were related to crown fracture (7.7%) (Fig. 7.5b), core fracture (7.7%) (Fig. 7.5c) and root fracture (7.7%) (Fig. 7.4h). The tooth, which showed a root fracture, had a screw-retained monolithic zirconia crown as an antagonist. The present results contrast with the literature. Bömicke et al. reported a 98.5% survival rate after 3 years in a prospective study of 82 monolithic crowns on natural teeth [16]. In that study, complications encountered also included the loss of retention and vertical root fracture. In another prospective study, Batson et al. reported a 100% survival rate for 10 monolithic second-generation zirconia crowns over 1 year [15]. The survival rate of the present study was also lower than the reported estimated 5-year survival rate of veneered zirconia, metal-ceramic, and lithium-disilicate reinforced glass ceramic crowns (96.0%, 94.7%, and 96.6%, respectively) [44]. In a systematic review by Sailer et al., crown fractures were less reported (0.4% for veneered zirconia crowns, 0.03% for metal-ceramic crowns, and 2.3% for lithium-disilicate reinforced glass ceramic crowns). Moreover, tooth fracture was predominantly found for metal-ceramic crowns (1.2%), and this complication occurred significantly less frequently for all ceramics. The higher rate of failures observed in the present study could be explained by the small sample size, which did not allow us to draw some significant conclusions, and by the inclusion of patients with high occlusal stress since most failures occurred in those patients (Fig. 7.2d). Indeed, in that case, the weak link can be the crown if the zirconia thickness is low, the core (core fracture), or the tooth (root fracture). The inclusion of such patients in clinical studies is crucial to validate techniques and procedures. Only one study included those patients and reported 1 crown fracture after 2 years in a case series including 84 crowns in 13 heavy grinders [28].

In the present study, the Kaplan-Meier success rate was only 81.8% ± 4.7% at 2 years (Fig. 7.3a). Prosthesis debonding (9.3%) impaired the success rate, but those failures were treated by performing a new bonding procedure. This event was anticipated for tooth-supported crowns as they were cemented with temporary cement for the study purposes. Implant-supported restorations debonding between zirconia crowns and titanium abutments were encountered only with RelyX Ultimate resin composite cement (28.0% of screw-retained restorations were cemented with RelyX Ultimate). For the new bonding procedure, Multilink Hybrid Abutment (Ivoclar Vivadent AG, Schaan, Liechtenstein) was preferred and showed better results.

The presence of implant loss, minor chipping (Fig. 7.5d), root as restoration fractures of antagonistic teeth, which were not considered in the success rate, must also be highlighted. One hypothesis is that zirconia's high stiffness and lack of resilience does not allow for occlusal stress absorption, as this stress is transferred to the implant, the tooth, or its antagonist. Indeed, most complications (76.9%) occurred in patients with bruxism clinical signs and only one regularly wore a nightguard as protection. This can support using a damping material when planning treatment in patients with bruxism to avoid any weak link effect [45,46]. Fig. 7.4 presents the history of a patient who showed unfavorable occlusal relationships and bruxism over 12 years. This clinical case illustrates the fact that improving material resistance does not necessary enhance treatment performance.

However, clinical FDI evaluations after 2 years were very good. Evaluators' assessments were high, even if a significant proportion of proximal contacts were judged as too weak. Experienced handling of the design software solved this issue. All of the biological aspects of the FDI criteria had high scores, showing the excellent biocompatibility of zirconia. Evaluators gave a lower score to aesthetic results than patients, who reported a high satisfaction rate. This result is consistent with other reports [15,16] and confirms that second-generation zirconia is appropriate for posterior restorations in terms of aesthetics.

Finally, the present study protocol allowed for wear quantification with laser profilometry performed directly on the restorations to avoid bias due to the use of replicas and for direct SEM observations to evaluate glaze wear in occlusal contact areas. Regarding wear, no zirconia wear was observable after two years, with respect to the accuracy and precision of the wear quantification experimental set up (15 µm). Laser profilometry and superimposition of scans is reputedly the best technique for clinical wear quantification [37], but unusable scans are often described, ranging from 14% to 31% [47–50]. Wear measurement was effective since quantification was conducted from the direct acquisition of the restoration surfaces and not from replicas as is frequently done for the measurement of antagonist tooth wear [17,20,30]. Indeed, the replica technique may explain the difference between the low zirconia wear reported in the present study. Cardelli et al. reported a 63 µm vertical loss when zirconia opposed enamel and a 19 µm vertical loss when zirconia opposed composite resin [17]. Glaze wear was highlighted on 70.2% of occlusal contact points areas after 6 months and on 100% after one year, which was predicted by Denry and Kelly [12]. Investigations regarding the influence of the finishing procedure showed that polishing should be preferred to glazing [51–60], which engenders a higher surface roughness and antagonistic tooth wear [61,62].

Limitations of the present study include the low number of FPDs (n = 14) and cemented crowns on teeth (n = 13), and the provisional cementation of those crowns due to study design.

5. CONCLUSION

In this prospective study, the global survival rate of the restorations was 93.3% after 2 years, FPDs having shown an excellent survival rate (100%) compared to crowns (95.8% for implant crowns and 76.9% for crowns on natural teeth), despite a high number of patients (61.7%) showing clinical signs of bruxism. The inclusion of patients with bruxism in clinical studies is rare but crucial to test the validity of new materials and techniques. It must be underlined that the sample size for implant FPDs (n = 14) and crowns on natural teeth (n = 13) was small and that further research is needed to confirm the present results. On the other hand, this study brings significant short-term data about 48 monolithic zirconia implant crowns, while the literature is sparse in that field and patients with bruxism were not examined. Zirconia wear was inferior to 15 µm after 2 years. Glaze wear was observed on all of the occlusal areas after 1 year, but not on the buccal and lingual/palatal faces. Clinical evaluation of the restorations showed good results after 2 years from aesthetic, functional, and biological perspectives. One crown fracture was reported. However, the treatment success rate after two years (81.8%) was not as high as expected. Eighty percent of the catastrophic failures and most of the complications (76.9%) occurred in patients with clinical signs of bruxism, who exhibited a not significantly lower restoration survival (92.3% versus 95.7%) and success (79.6% versus 87.0%) rates at two years. The present results underscore, from the authors' point of view, a weakness of full zirconia restorations, which are strong but stiff and unable to absorb stresses. This could be suspected to promote the "weak link theory", that is, a breaker effect on the weaker parts of the system, which can be the bonding interface, the supporting tooth, the supporting implant, or the antagonistic teeth/implants. Consequently, single-unit full zirconia restorations should be used with caution until further and long-term research is conducted considering samples, which do not exclude bruxers.

6. DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

7. ACKNOWLEDGMENTS

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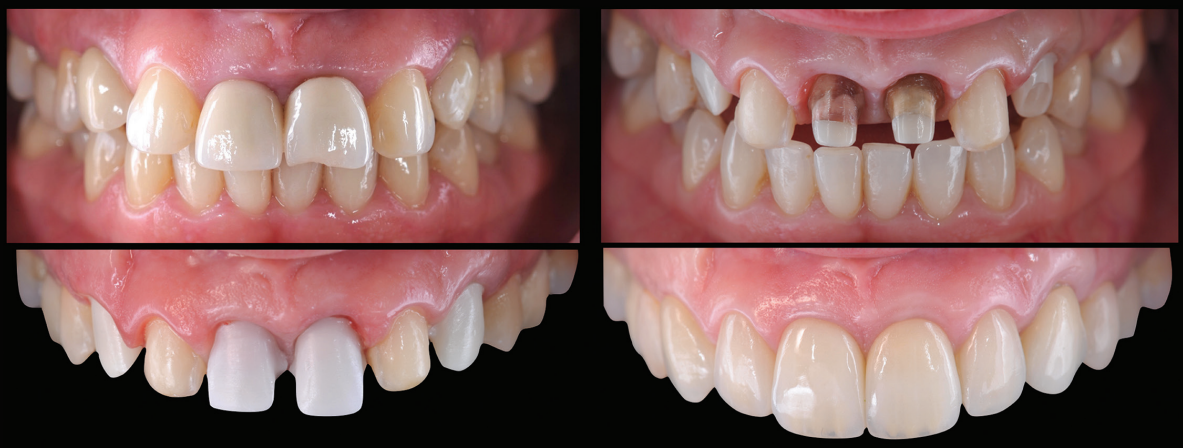

8 Discussion and conclusions

1. DISCUSSION

1.1. Main study protocol

It must be noticed that the evaluation of antagonistic teeth wear, and then zirconia abrasive effect, could not be measured. Indeed, as explained in the systematic review about clinical wear measurement, the use of replica significantly reduced the accuracy of the scans and their superimposition process. Therefore, it was decided not to use those results.

1.2. From 1st to 3rd generation zirconia: clinical considerations based on research outcomes

Nowadays, dental zirconia constitutes a large family of materials with various compositions and properties. At the beginning of this research work, monolithic zirconia restorations in second-generation 3Y-TZP were not widespread, and high-translucency zirconia (3rd generation) did not exist yet. Veneered zirconia restorations in **first-generation zirconia** were mostly used, and there was a long learning curve to reduce fracture problems, particularly chipping of the veneering ceramic, which was shown to be more frequent than with the gold standard, that is, porcelain-fused to metal (PFM) systems. Today, practitioners and dental technicians pay much more attention to several risk factors related to prosthesis manufacturing, which have been shown to promote this problem. The first parameter, which has been changed, is the design of the framework. Indeed, at the beginning of the use of computer-aided design and manufacturing (CAD/CAM) processes, this design was not supportive of the veneering ceramic, the frameworks being simple copings of tooth preparation. Various recommendations have been formulated to reduce residual stresses in the veneering ceramic and improve its mechanical resistance, such as minimizing the thickness of the veneering ceramic and

increasing the thickness of the frameworks (Inokoshi et al. 2016; Mainjot et al. 2015; Mainjot et al. 2011; 2012a; 2012b; Silva et al. 2011).

The first part of this work introduced another type of risk factor besides the material manufacturing process: **the patient and the associated clinical risk factors** (Koenig et al. 2013). Indeed, the results proved that patients with clinical signs of bruxism are much more at risk of chipping and that wearing a nightguard significantly prevents failure. In addition, the presence of ceramic restorations as an antagonist, implant-supported restorations (due to the absence of stress resilience via the periodontal ligament), and restorations with a high number of elements (due to the unfavorable distribution of the stresses) need to be considered by the dentist as situations with higher failure rates. Recent clinical studies indicate that chipping remains the predominant technical complication of veneered zirconia and is still more frequent than with PFM in the case of bridges, but the failure rate is similar for zirconia and PFM crowns, which is promising (Pjetursson et al. 2018; Sailer et al. 2018).

First-generation zirconia remains advantageous in terms of mechanical resistance, constituting the only ceramic materials capable of satisfying the indications for posterior and/or long-span bridges. It is also very suitable for tooth-supported prostheses, especially for masking a colored abutment or reproducing a high-value tooth. Owing to its high biocompatibility, zirconia is also an ideal material for implant-supported prostheses, and it advantageously replaces titanium for abutments and PFM restorations for both anterior and posterior restorations. When possible, screw-retained restorations should be preferred to obtain a very favorable ratio between the thickness of the framework and the veneering ceramic. Moreover, the advent of digital technologies offers the possibility of making in advance, before the placement of the implant, a custom-made implant abutment designed ideally to the desired emergence profile (Figure 8.1). This abutment and the provisional crown are placed on the day of surgery for immediate loading, following the "one abutment-one time" concept. It allows soft tissue healing directly on the

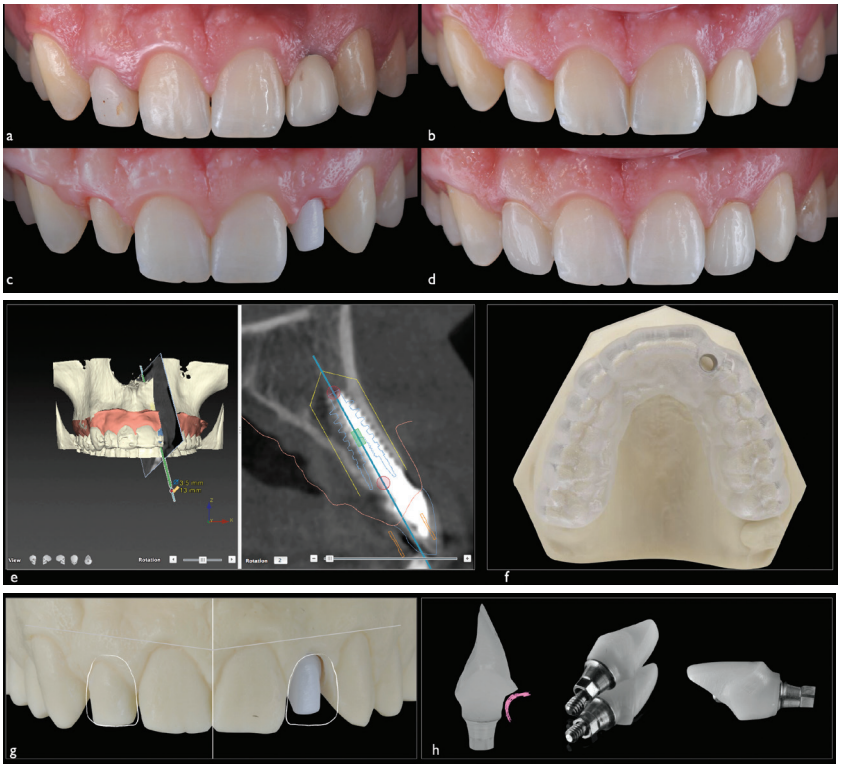

Fig 8.1 *Figure 8.1: Zirconia abutment (NobelProcera, Nobel Biocare) on a bone level implant (NobelActive NP), custom made before implant placement on the basis of the digital planning of the surgery (NobelClinician, guided surgery with Nobelguide, Nobel Biocare).*

a) View before treatment: the patient has a provisional implant on #22.
b) View one week after extraction of the provisional implant, placement of the implant, connective tissue graft, placement of the abutment and the temporary crown in one step.
c) View of the abutment and the preparation of #12 for a veneer (conoid tooth).
d) View after bonding of lithium disilicate reinforced glass-ceramic restorations (IPS e.max Press, Ivoclar Vivadent).
e) View of the digital planning.
f) Surgical guide.
g) h) Design of the abutment on the model, following esthetic analysis and an ideal emergence profile including a concave transgingival part. Prosthodontics: Prof Amélie Mainjot. Surgery: Prof France Lambert. Dental lab: Dental team, Luc and Patrick Rutten, Belgium. From "Zircone(s) Partie 1 - A la rencontre de céramiques pas comme les autres ", Mainjot A, in Biomatériaux Cliniques, Vol 3, n°1, mars 2018.

zirconia, according to the ideal emergence profile, and prevents subsequent rupture of the cell attachment via unscrewing. The abutment is associated with a titanium base (ASC abutment, Nobel Biocare) which ensures the connection with the implant.

The **second-generation zirconia** is a promising material for realizing high-strength monolithic or partially veneered posterior restorations (Figure 8.2), both

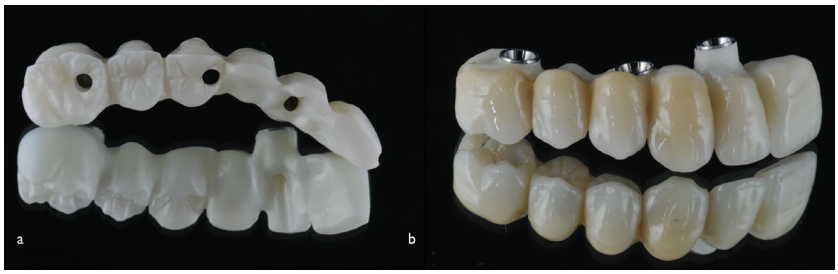

Fig 8.2 *Second-generation zirconia screwed-retained FPD (Prettau 2 dispersive, Zirkonzahn, Italy): in this case, to improve the aesthetic result, a part of the prosthesis has been veneered.*
a) Framework designed to veneer the buccal part.
b) After veneering of the buccal part. Dental lab: Dental team, Luc and Patrick Rutten, Belgium. From "Zircone(s) Partie 2 – Restaurations émaillées ou monolithiques ? La quête de la résistance et de la translucidité ", Mainjot A, in Biomatériaux Cliniques, Vol 4, n°2, octobre 2019.

single and plurals; however, contrary to manufacturers' recommendations, caution is required in patients with bruxism in the attempt of long-term clinical follow-up on their biomechanical behavior, and the wearing of a nightguard can be recommended to accommodate the stresses in these patients. It must also be noted that the observed low wear rate of zirconia in comparison with tooth tissues requires regular occlusal contact checking in order to avoid the appearance of high occlusion on prostheses with time. Concerning the impact of the low-temperature degradation (LTD) process described in this work, it is unknown and must be explored in the long term since these zirconia are more sensitive to it than the 1st generation. The observed chipping and major fractures in this study, as in others (Habibi et al. 2020; Hansen et al. 2018; Miura et al. 2020) were not expected with such high-strength zirconia materials. The LTD images shown in this work are interestingly similar to images obtained

in explanted zirconia orthopedic prostheses, and this phenomenon could explain the observed fractures, particularly in patients with high occlusal stress, which can accelerate LTD. Moreover, LTD and tribological stresses generate a pull-out of monoclinic grains, which are released in the body and could constitute a point of concern. Indeed, the wide application of nanoparticles has raised an issue on their potential risk for health and environment. Up to now, toxicological studies on zirconium oxide (ZrO^2) nanoparticles are limited, and the results are controversial. Some studies reported that ZrO^2 nanoparticles can induce mild (Karunakaran et al. 2013; Lanone et al. 2009) or no cytotoxic effects (Dalal et al. 2012; Demir et al. 2013; Soto et al. 2007). Other authors underlined that ZrO^2 nanoparticles have better biocompatibility properties than other nanomaterials such as ferric oxide, titanium dioxide or zinc oxide (Brunner et al. 2006; Karunakaran et al. 2013; Landsiedel et al. 2014; Otero-González et al. 2013), while Ye and Shi indicated that ZrO^2 nanoparticles can induce toxic effects at high concentrations, affecting cell viability, inducing apoptosis and necrosis, or changes in cell morphology (Ye and Shi 2018).

Finally, the **third-generation zirconia** is significantly more translucent but also significantly less mechanically resistant and cannot achieve such broad indications as the other two generations. Moreover, its behavior in terms of LTD should be studied since those materials still contain a significant proportion of tetragonal phase. If manufacturers recommend it for 3-unit bridges, it remains risky (Figure 8.3) and its use may be limited to single-unit elements. No clinical knowledge is currently available on this type of material, which is also recommended by manufacturers for resin-bonded partial restorations such as veneers, but cannot exhibit the same bonding properties to resins as glass-ceramics. For these reasons, glass-ceramics remain the gold standard for the moment.

Finally, it must be highlighted that practioners and dental technicians should be better informed about the type of zirconia material they use in terms of

composition (and then properties), which is difficult with respect to the lack of data delivered by manufacturers.

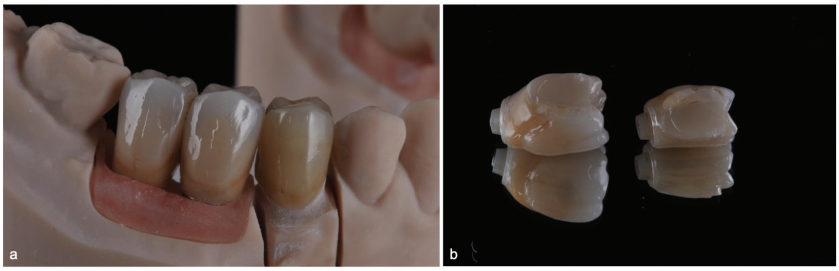

Fig 8.3 *a) Screw-retained 2-unit FPD in third-generation zirconia (Prettau 4 anterior dispersive, Zirkonzahn, Italy) on #45 and #45 bis.*
b) Fracture of the connection area during the try-in of the prosthesis.

1.3. Does mechanical strength warrant clinical success?

The originality of this work is the fact that, unlike almost all of the available clinical studies on monolithic restorations, patients with clinical signs of bruxism were included and represented nearly 61% of the sample in the main study. The clinical signs of bruxism were registered with a standard non-instrumental approach of bruxism diagnosis, including a clinical inspection and self-report, notably following the criteria of the American Academy of Sleep Medicine (American academy of sleep disorders, international classification of sleep disorders 2014; Casett et al. 2017; d'Incau 2017). However, bruxism diagnosis is complex and inaccurate, and if the employed criteria are graded 2 on 3 in terms of assessment validity (meaning "probable sleep/awake bruxism"), polysomnography is recommended to confirm diagnosis (Lobbezoo et al. 2018), which was not possible in this large-scale clinical study. All of the patients recorded as bruxers presented abnormal attrition wear facets and reported teeth grinding during the night or day, which means that the presence of bruxism is probable but can engender some false positives (Casett et al. 2017). Consequently, it can explain the high proportion of patients considered bruxers compared to the literature data, but those

data are restricted, particularly for awake bruxism, and diagnostic criteria are greatly heterogeneous (reported prevalence is 22-30% for awake bruxism and 1-15% for sleep bruxism) (Melo et al. 2019). It must also be noticed that the bruxism diagnosis should be repeated during the study because this behavior is not necessarily continuous (Kato et al. 2013). The inclusion of patients with bruxism in clinical studies is rare but crucial for testing the validity of new materials and techniques in unfavorable situations. In addition, clinical examination included observation of antagonistic teeth. The results at the 2-year follow-up highlighted worrying short-term failures related to implant loss (2.7%), root fractures of the supporting teeth (1.3%), antagonistic teeth (1.6%), and composite fractures present on the antagonistic teeth (1.6%). In comparison, Oudkerk *et al.* (Oudkerk et al. 2020) studied the performance of bonded CAD-CAM polymer-infiltrated ceramic network materials (PICN) restorations in patients with severe tooth wear and bruxism after two years. Surprisingly, they showed better results than in the present study about zirconia restorations, with a survival rate of 100% and a success rate of 93.8 % (versus 93.3% and 81.8% in the present work, respectively), and this despite the lower mechanical resistance of PICN material and the extreme conditions to which it was subjected.

In fact, if zirconia materials are clearly more resistant than all other dental ceramic and composite materials, they may not perform better in high-risk patients, probably because of the high stiffness of all zirconia materials (approximately 210 GPa), which are not able to accommodate occlusal stresses by elastic or plastic deformation, and transfer it to substructures, that is, the supporting tooth or implant, or the antagonistic teeth.

Indeed, if occlusal stresses are too high, there will always be a weak link in the system, which will play the role of a breaker. With first-generation zirconia, this weak link was the veneering ceramic, and with monolithic restorations, it could be the supporting tooth, implant, or antagonistic teeth (Figure 8.4). Consequently, for patients with bruxism clinical signs, it may be advisable to

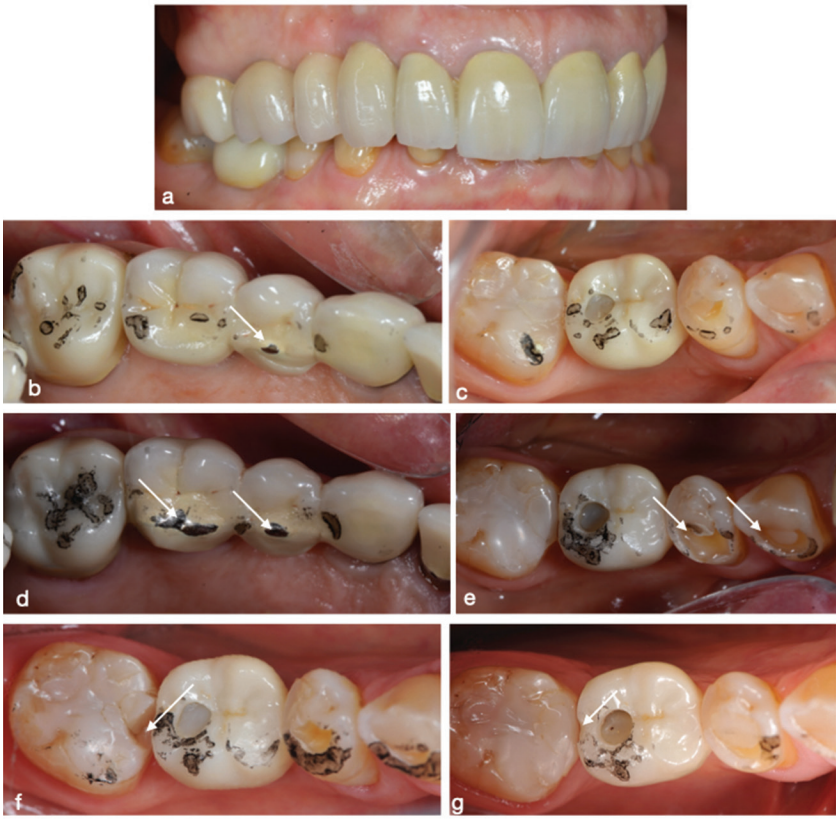

Fig 8.4 *Illustration of the weak link theory, with failures in a patient presenting bruxism clinical signs and not wearing a nightguard.*
a) Full zirconia cemented crown (tooth #17) and full zirconia screw-retained crown (tooth #46).
b) Occlusal view (tooth #17) at baseline. Chipping previously present on pontic #14 (screw-retained PFM FPD).
c) Occlusal view (tooth #46) at baseline.
d) Veneering chipping on an antagonistic tooth (tooth #16) at the 1-year follow-up.
e) Composite adhesive fracture on antagonistic teeth #44 and #45 at the 1-year follow-up.
f) Composite chipping on an antagonistic tooth #47 at the 2-year follow-up.
g) Minor chipping (tooth #46) at the 2-year follow-up.

favor the use of CAD-CAM composite materials, which are less resistant to flexural strength than zirconia but are more able to accommodate stress due to their polymeric matrix (Figure 8.5).

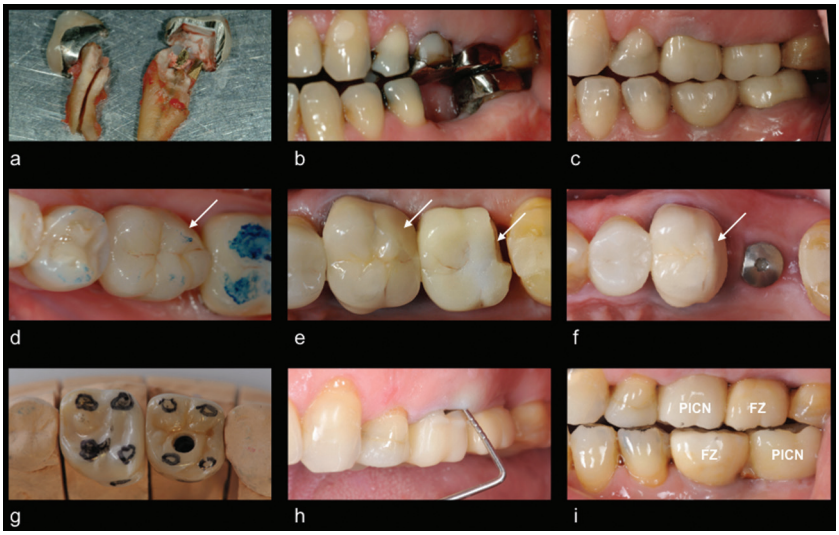

Fig 8.5 *Patient.*
a) and b) Root fracture #36 and extraction.
c) Realization of veneered zirconia crowns on teeth #26, #27, and #37 and implant #36.
d) and e) Chipping (arrowed) on #26 and #36 after 6 months and on #27 after 3 years.
f) Root fracture #27 after 8 years.
g) Monolithic zirconia crowns #26, #27, and #36.
h) Root fracture on tooth #26 after 1 year.
i) Use of polymer-infiltrated ceramic network (PICN) material for crowns on implant #26 and implant #37 at a 2-year follow-up. Full zirconia (FZ) restorations on implant #27 and implant #36 at a 3-year follow-up. From (Koenig et al. 2019).

One of the major conclusions of this work is that the mechanical resistance of prosthodontic materials, which has always driven the development strategies of dental manufacturers, does not warrant the clinical performance of treatments. In fact, the idea is to consider the entire system from a biomechanical point of view, and not only the prosthesis itself. In this context, regardless of the material choice, the patient and the related clinical parameters, particularly the presence of bruxism, are major influencing factors of treatment success rate.

The quest for mechanical resistance takes us away from the ideal of a biomimetic biomaterial, that is, the concept of replacing missing tissues with a biomaterial showing similar properties to enamel and dentin, respectively.

For example, similar gradients in elastic modulus and similar wear properties have been shown in some experimental PICN (Eldafrawy et al. 2018). The treatment objective is the survival of the prosthesis support, whether it is a tooth or an implant, as well as that of the antagonistic teeth. Unfortunately, the ideal biomaterial does not exist yet, and there is no universal biomaterial that can meet all indications. The practitioner must make an informed choice according to the clinical situation, without sacrificing treatment quality for economic benefit, which is tempting knowing that low-cost full zirconia restorations have now entered the market.

2. CONCLUSIONS AND PERSPECTIVES

This work highlights two issues that must be monitored and followed over time: the LTD of the material and the global biomechanical behavior of the system prosthesis-supporting tooth or implant, particularly in high-risk patients with bruxism.

The main perspectives of this work are certainly the expected long-term results of the principal clinical study of this thesis. The original approach of the protocol, which allows for wear quantification with 3D laser profilometry performed directly on the restorations to avoid bias due to the use of replicas and for *in vitro* investigation of material aging with time, will furnish further precious data on the *in vivo* LTD of biomedical 3-YZP zirconia, and on the effect of mechanical/tribological stress on this process. The advantage of dental applications is that, contrary to orthopedics, the prostheses can be temporarily removed from the oral cavity to perform *ex vivo* analyses and provide a qualitative and quantitative evaluation of LTD. In this context, this work has shown for the first time that *m*-phase quantification using XRD or Raman spectroscopy can underestimate the LTD process, particularly in areas subjected to tribological stress because of grain pull-out.

Future perspectives include the use of high-performance modern techniques to monitor LTD, such as Raman mapping. They also comprise a thorough analysis of fractured restorations (chipping and major fractures), for example, with high-performance scanning electron microscopy imagery and atomic force microscopy, and probably cross-sectioning samples or using focused ion beam in order to study the LTD in-depth penetration and its influence on crack initiation and propagation.

The long-term results of the study will also provide crucial information on the long-term general clinical performance of monolithic zirconia prostheses, particularly in high-risk patients, which were shown in this thesis to be bruxers; this population of patients is important and rarely included in clinical studies. Since it was shown that the short-term failure rates are higher in those cases, long-term results will answer the question of the appropriateness of using monolithic zirconia prostheses in bruxers, particularly with respect to failures of supporting teeth, implants, or antagonistic teeth, which could be accentuated by the high stiffness of the material and its inability to accommodate occlusal stress. Indeed, contrary to marketing arguments developed by companies, this study shows that the mechanical strength of the material does not constitute a warranty of clinical success. The prosthesis is just a link in a system, which should promote global biomechanical behavior, and the preservation of the teeth of the patients for all their lives.

Finally, the world dental market of zirconia is booming, representing around 116 million dollars in 2020, and is expected to increase by 50% by 2025 (Global zirconia dental material market 2020 by manufacturers, regions, type and application, forecast to 2025. 2020). This means that a significant amount of zirconia crowns and bridges are placed in patients every year and that this amount will increase in the future, and the long-term clinical behavior of these prostheses is still unknown. Unfortunately, the biomaterials history shows that products are often put on the market by manufacturers before sufficient knowledge is available on their properties, particularly in terms of

aging, which has already led to catastrophic clinical failures, such as those encountered with hip prostheses. In this context, this work shows that LTD exists in dental prostheses and that the impact of this phenomenon is currently unknown and is not under control. It should be considered to be able to define the limits of zirconia use and to warranty long-term satisfaction in our patients.

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ASSOCIATED STUDENT WORK

Y-TZP: In-mouth aging and low-temperature degradation: A 5-year prospective clinical study with Lava Plus® restorations. Thesis presented by Caroline Pepinster to obtain Master degree in Dental Sciences, 2016, University of Liège (ULiège).

Y-TZP: Aging and low-temperature degradation: Contribution to wear analysis in a 5-year prospective clinical study using Lava Plus® restorations. Thesis presented by Guillaume Martin to obtain Master degree in Dental Sciences, 2017, University of Liège (ULiège).

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POSTERS AND ORAL COMMUNICATIONS IN INTERNATIONAL CONGRESSES

Posters (abstracts published)

In Vivo low-temperature degradation of monolithic zirconia restorations. V. Koenig, C. Wulfman, N. Dupont, S. Bekaert, S. Le Goff, M. Eldafrawy, G. Martin,

T. Douillard, J. Chevalier, A. Vanheusden, A. Mainjot. European Dental Materials Conference (EDMC), Brussels, Belgium, August 2019.

In vivo aging of second-generation monolithic zirconia restorations. V. Koenig, C. Wulfman, N. Dupont, S. Bekaert, S. Le Goff, M. Eldafrawy, G. Martin, T. Douillard, J. Chevalier, A. Vanheusden, A. Mainjot. French speaking Society of Dental Biomaterials (SFBD) congress, Paris, France, July 2019.

In vivo low thermal degradation of monolithic zirconia restorations. C. Wulfman, V. Koenig, N. Dupont, S. Bekaert, S. Le Goff, M. Eldafrawy, G. Martin, A. Vanheusden, A. Mainjot. Academy of Dental Materials (ADM) meeting, Nuremberg, Germany, October 2017.

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Oral communications (abstracts published)

Three-year Clinical Results of Second-Generation Zirconia Monolithic Posterior Restorations. V. Koenig, S. Bekaert, N. Dupont, S. Le Goff, A. Vanheusden, C. Wulfman A. Mainjot. International Association for Dental Research (IADR)

Continental Europe Division meeting, Brussels, Belgium, September 2021 (accepted).

In vivo low-temperature degradation of monolithic zirconia restorations. IADR general session meeting, London, England, July 2018.

Low thermal degradation of monolithic zirconia dental prostheses: 1-yr results of a prospective clinical study with *ex vivo* analyses. V. Koenig, C. Wulfman, N. Dupont, S. Bekaert, S. Le Goff, M. Eldafrawy, G. Martin, A. Vanheusden, A. Mainjot. SFBD congress, Paris, France, June 2017.

Comportement clinique des couronnes monolithiques en zircone. Les Entretiens de Bichat, Paris, France, October 2016.

Clinical risk factors related to failures with zirconia-based restorations: An up to 9-year retrospective study. Espertise Talent Award, Seefeld, Germany, November 2013.

CURRICULUM VITÆ

Vinciane Koenig was born in Liège, Belgium, on August 23, 1986. She is married and has two daughters, Eva (born in 2014) and Lise (born in 2016).

She received her DDS degree from the University of Liège (Belgium) in 2009. From 2009 to 2012, she completed a full-time post-graduate program in Oral Rehabilitation in the same university, and graduated *Summa Cum Laude*. She has a clinical activity three days a week as Head of Clinic in the department of Fixed Prosthodontics at the University Hospital of Liège (Head: Prof. A. Vanheusden) and is also involved in pre-doctorate education, supervising clinical internships of dental students. She has clinical expertise in the field of restorative dentistry and fixed prosthodontics, particularly minimally invasive treatments and multidisciplinary management of complex rehabilitation cases on teeth and implants. She is a PhD student in the dental-Biomaterials Research Unit (d-BRU) at the University of Liège (Head: Prof. A. Mainjot), where she has a research activity in close collaboration with the University of Paris, France (Prof. C. Wulfman). Her scientific interest is dental ceramics, particularly zirconia.

CLINICAL BEHAVIOR AND INTRAORAL LOW-TEMPERATURE DEGRADATION OF ZIRCONIA DENTAL PROSTHESES

The use of zirconia in the field of dental prostheses has grown significantly since its introduction in the 2000s, following the advent of computer-aided design and manufacturing technologies. First-generation zirconia-based restorations (ZBR) are bilayered structures composed of a framework and a glass-ceramic veneer, which imparts an essential esthetic appearance. However, the first clinical reports regarding veneered ZBR indicated a high rate of short-term failures due to cohesive fractures (chipping) of the veneering ceramic. To address this, a retrospective study on veneered ZBR was conducted to investigate the influence of clinical parameters, such as patient-related risk factors, on chipping failures.

Zirconia is now used to fabricate monolithic dental prostheses without the veneering ceramic layer and the only presence of a thin cosmetic glaze. These prostheses were notably developed to remedy chipping and, thus, obtain prostheses with an increased durability. Therefore, zirconia materials exhibiting greater translucency have been developed for monolithic restorations However, second-generation zirconia materials are prone to show greater metastable behavior, which could promote low-temperature degradation (LTD).

The main objective of this study was to evaluate the intraoral LTD of zirconia monolithic restorations and the influence of occlusal stresses and glaze protection on this process. Secondary objectives included the investigation of the general clinical behavior and material wear of the restorations. This work introduces an original protocol, including *ex vivo* analyses, to evaluate the LTD process of monolithic zirconia prostheses in the oral environment and to study their general clinical behavior, primarily in terms of material wear.

LTD was shown to develop in 3 mol% yttria-doped tetragonal zirconia polycrystal monolithic restorations six months after intraoral placement and then progresses over time. After two years, the tetragonal-monoclinic transformation became non-uniform, with the presence of localized clusters of transformed grains. In axial areas, the grain aspect was typical of the classical nucleation-growth process reported for LTD. However, in occlusal areas, tribological stress generated surface crushing and grain pull-out from the clusters, causing an underestimation of the aging degree when the evaluation was limited to monoclinic phase quantification. Glazing cannot be considered a protection against LTD because it is worn away in occlusal areas after one year.

Two years after their implantation, the Kaplan-Meier survival rate of restorations was 93.3% (100% for fixed partial dentures) and the success rate was 81.8%. It was found that eighty percent of major failures occurred in patients exhibiting clinical signs of bruxism. Complications such as root fracture, periodontal disease or composite chipping were also observed in antagonistic teeth. Wear in zirconia was observed to be less than 15 µm. The success rate of single-unit restorations was not as high as expected, the weak link being the prosthesis support or the antagonist tooth.

One hypothesis is that zirconia's stiffness and lack of ability to deform do not promote occlusal stress accommodation, which can be critical in patients affected by bruxism. Furthermore, several unexpected clinical failures were observed, including chipping and major fracture of the material, which may have been induced through LTD. If LTD occurs through the same mechanisms in dental prostheses as in orthopedic prostheses, its clinical impact remains unknown and needs to be evaluated through a thorough analysis of fractured prostheses in the framework of long-term studies.

Keywords
Computer-aided design/computer-aided manufacturing, Raman spectroscopy, *t-m* transformation, Aging, Biomaterial, Dental prosthesis, Dental implant, Wear measurement, Ceramic, Fracture, Clinical study, *Ex vivo* analysis.

Vinciane KOENIG

45 Quai G. Kurth, 4020 Liège, Belgium
tel : +32 4 270 31 06
email : vinciane.koenig@chuliege.be