Phase transformation and fracture load of stock and CAD/CAM-customized zirconia abutments after 1 year of clinical function
Schepke, Ulf; Gresnigt, Marco M M; Browne, Wesley R; Abdolahzadeh, Shaghayegh; Nijkamp, Joey; Cune, Marco S

Published in: CLINICAL ORAL IMPLANTS RESEARCH

DOI: 10.1111/clr.13442

IMPORTANT NOTE: You are advised to consult the publisher's version (publisher's PDF) if you wish to cite from it. Please check the document version below.

Document Version
Publisher's PDF, also known as Version of record

Publication date: 2019

Link to publication in University of Groningen/UMCG research database

Citation for published version (APA):

Copyright
Other than for strictly personal use, it is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license (like Creative Commons).

Take-down policy
If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Downloaded from the University of Groningen/UMCG research database (Pure): http://www.rug.nl/research/portal. For technical reasons the number of authors shown on this cover page is limited to 10 maximum.

Download date: 16-01-2020
Phase transformation and fracture load of stock and CAD/CAM-customized zirconia abutments after 1 year of clinical function

Ulf Schepke1 | Marco M. M. Gresnigt1 | Wesley R. Browne2 | Shaghayegh Abdolahzadeh2 | Joey Nijkamp1 | Marco S. Cune1,3,4

Abstract

Objectives: Functional loading and low-temperature degradation may give rise to impaired clinical long-term service of zirconia implant abutments. The aim of this study was to compare the fracture strength (primary outcome measure) and the volume percentage of monoclinic surface zirconia (m-ZrO₂) of stock and CAD/CAM-customized zirconia implant abutments that functioned clinically for 1 year with geometrically identical pristine controls in an ex vivo experiment.

Material and methods: Twenty-three stock (ZirDesign™) and 23 CAD/CAM-customized (Atlantis™) zirconia implant abutments were retrieved after 1 year of clinical service. They were compared with pristine copies with respect to the volume fraction of the monoclinic phase using Raman spectroscopy and their fracture load by means of a single load-to-fracture test. Failure analysis was performed using optical and SEM microscopy. After verification of normal distribution, paired t tests were used for comparison of fracture loads between pristine and clinically aged specimen. All statistical tests employed a level of significance of α = 0.05.

Results: The fracture loads of the stock zirconia abutments were significantly (p < 0.05) reduced to 78.8% (SD 29.5%) after one year of clinical function. For the CAD/CAM abutments, no reduction in fracture load was found. No m-ZrO₂ volume percentages beyond the detection threshold of 5% were observed in any of the samples.

Conclusions: After 1 year of clinical service, no difference in fracture strength of the CAD/CAM-customized zirconia implant abutments could be demonstrated, whereas the stock zirconia abutments decreased considerably in fracture strength. No substantial tetragonal-to-monoclinic transformation was observed.

KEYWORDS
biomaterials, clinical research, clinical trials, material sciences, prosthodontics, surface chemistry
Results from in vitro experiments indicate that modern high-strength ceramics such as yttria-stabilized tetragonal zirconia polycrystal (zirconia) should be able to withstand physiologic, functional, occlusal forces (Adatia, Bayne, Cooper, & Thompson, 2009; Aramouni et al., 2008; Att, Kurun, Gerds, & Strub, 2006; Coray, Zeltner, & Ozcan, 2016; Yuzugullu & Avci, 2008). The material and design of an abutment reflect on its mechanical aspects.

In general, titanium (Ti) abutments with internal, conical connections are stronger than abutments with an external, hexagonal connection and stronger than zirconia abutments. Hybrid zirconia abutments with a secondary metallic ring are stronger than one-piece, monolithic zirconia abutments (Alsahaf, Spies, Vach, & Kohal, 2017; Chuen et al., 2015; Elsayed, Wille, Al-Akhal, & Kern, 2017; Foong, Judge, Palamara, & Swain, 2013; Gehrke, Johansson, Fischer, Stawarczyk, & Beuer, 2015; Leutert, Stawarczyk, Truninger, Hammerle, & Sailer, 2012; Martinez-Rus, Ferreiroa, Ozcan, Bartolome, & Pradies, 2012; Mitsias, Thompson, Pines, & Silva, 2015; Muhlemann, Truninger, Stawarczyk, Hammerle, & Sailer, 2014; Sailer, Sailer, Stawarczyk, Jung, & Hammerle, 2009b; Sghaireen, 2015; Truninger et al., 2012; Zendparsa & Albosefi, 2016). A Ti interface induces less wear to the Ti internal implant body than when a monolithic abutment is used (Cavusoglu, Akca, Gurbuz, & Cehreli, 2014; Klotz, Taylor, & Goldberg, 2011; Stimmelmayr et al., 2012). Differences in mechanical strength between geometrically identical stock and CAD/CAM-customized abutments are not observed (Gehrke et al., 2015). Small diameter zirconia abutments are less resistant to static loads than abutments with greater diameters (Shabanpour, Mousavi, Ghodsi, & Alikhani, 2015). Angulated zirconia abutments are more prone to fracture than straight ones (Albosefi, Finkelman, & Zendparsa, 2014; Thulasidas et al., 2015). Fracture behavior of a zirconia abutment is also dependent on whether or not the abutment is restored (Muhlemann et al., 2014) and by the geometry of the crown (Nothdurft, Neumann, & Knauber, 2014). Finally, preparation of zirconia abutments does not reduce its fracture resistance significantly (Adatia et al., 2009).

The few clinical studies with medium- to long-term results that are available present acceptable results for various abutment types. Failure rates and incidence rates of technical complications appear similar for ceramic and titanium abutments (Canullo, 2007; Cooper, Stanford, Feine, & McGuire, 2016; Glauer et al., 2004; Hobkirk et al., 2009; Passos, Linke, Larjava, & French, 2016; Rinke, Lattke, Eickholz, Kramer, & Ziebolz, 2015; Sailer et al., 2009; Zemic, Bosch, Jung, Hammerle, & Sailer, 2013; Zemic, Philipp, Hammerle, Wohlwend, & Sailer, 2015). However, fracture of zirconia abutments is both anecdotic and reported in the literature (Aboushelib & Salameh, 2009; Joda & Bragger, 2015; Passos et al., 2016).

Concerns have been raised with respect to the reduction in strength of zirconia materials in time. It has been demonstrated that cyclic loading causes a reduction of fracture strength of various abutment types (Coray et al., 2016). In addition, it is hypothesized that detrimental tetragonal-to-monoclinic phase transformation occurs resulting from mechanical stresses or more spontaneously, as a consequence of hydrothermal degradation, also described as low-temperature degradation (LTD) or aging (de Basilio et al., 2016; Chevalier, Cales, & Drouin, 1999; Lugi & Sergio, 2010). Accelerated aging induces LTD at the surface and concomitant increased roughness. This in turn facilitates the initiation of micro-cracks, which in the laboratory may or may not contribute to a decrease in strength (Alghazzawi et al., 2012; Lucas, Lawson, Janowski, & Burgess, 2015).

A shift in volume percentage of monoclinic zirconia (m-ZrO_2) can be interpreted as aging of the material and serve as an indirect indication of the reduction in strength of the bulk. ISO 13356:2015 sets the acceptable norm for m-ZrO_2 in when used as implants for surgery at a maximum of 25% after accelerated aging, which could serve as some kind of reference (International Organisation for Standardisation, 2015).

From a consensus report (Hobkirk et al., 2009) based on the results of a systematic review (Sailer et al., 2009), standardisation of strength tests was advised, and since then, more in vitro studies in the literature abide by the applicable standards set by the International Organisation of Standardisation (ISO 1099:2017 and 14801:2016) on loading of the dental implant–abutment assembly (International Organisation for Standardisation, 2016, 2017). These standards do not include accelerated, artificial hydrothermal aging of the ceramic abutments which, in laboratory studies, is achieved in different ways. Ambivalent data are reported with respect to the effect of such hydrothermal challenges on zirconia substrates (Basilio et al., 2016; Nothdurft et al., 2014; Pereira, Muller, et al., 2016; Pereira, Venturini, et al., 2016). Studies in which loading is achieved under more realistic, preferably clinical conditions are likely to present with substantially more external validity.

The aim of the present study was to compare the fracture strength (primary outcome measure) and the volume percentage of surface m-ZrO_2 (secondary outcome measure) of stock and CAD/CAM-customized zirconia implant abutments that functioned clinically for 1 year with geometrically identical pristine controls. The null hypotheses are that the fracture strength of a zirconia abutment and the volume percentage m-ZrO_2, be it stock or CAD/CAM-customized, do not deteriorate significantly and that the percentage of m-ZrO_2 does not exceed the ISO 13356:2015 norm of 25% after 1 year of clinical function.

2 MATERIAL AND METHODS

2.1 Study design

A single-center, randomized controlled clinical trial was designed. Participants could participate if they were missing a single mandibular or maxillary premolar. Inclusion and exclusion criteria are listed in Table 1.

Permission from the medical ethics committee of the university medical center Groningen, The Netherlands, was granted (METc
number 2012.388, ABR number NL 42288.042.12), and informed consent was obtained. Primary outcome measure was the relative reduction in mean fracture strength upon static loading between pairs of pristine zirconia abutments and abutments that had functioned clinically for a year, be it stock and CAD/CAM-customized.

### 2.2 | Sample size estimation

Data from an in vitro study by Truniger et al. (2012) provide fracture strengths of zirconia abutments that we aim to use (internal hexagon, zirconia matching part) being 332 N (SD 58 N) and for another type of zirconia abutment (internal hexagon, titanium matching part) being 380 N (SD 59 N). We presume that non-aged zirconia abutments without titanium matching part will be somewhere in between these two values, which we chose to set at 360 N (approximately 5% less than zirconia abutments with a titanium matching part). Based on these numbers we estimated that a reduction of 10% in fracture strength would be a reasonable assumption for ageing, yielding an effect size of 0.6 (assumed SD 58.5, assumed difference 36 (=10% of 360 N). We further slightly reduced the effect size to 0.5 to not to overestimate the effect being an medium effect size (Cohen, 1988). Alpha was set at 0.05 for one-sided testing, and in conjunction with a desired power of 0.90, the a-priori sample size was calculated to be 36 abutments. (G-Power 3.1.9.2 Software, University of Dusseldorf; Faul, Erdfelder, Lang, & Buchner, 2007). Because we feel that some other outcome measures may be less discriminative and to allow for dropouts, we included 50 patients, 25 per group.

### TABLE 1  Inclusion and exclusion criteria

<table>
<thead>
<tr>
<th>Inclusion criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>Missing a first or second premolar in the maxilla or mandible</td>
</tr>
<tr>
<td>Desire to replace the missing premolar with an implant</td>
</tr>
<tr>
<td>Willing to sign for informed consent</td>
</tr>
<tr>
<td>Bone height ≥10 mm beneath the maxillary sinus and</td>
</tr>
<tr>
<td>≥10 mm above the mandibular nerve and a bone width of at least 6 mm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Exclusion criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>Missing teeth mesial or distal from implantation site</td>
</tr>
<tr>
<td>Orthodontic treatment at the time of impression taking</td>
</tr>
<tr>
<td>Severe bruxism</td>
</tr>
<tr>
<td>Acute periodontitis</td>
</tr>
<tr>
<td>History of implant loss</td>
</tr>
<tr>
<td>Documented extreme gagging reflex</td>
</tr>
<tr>
<td>Poor medical condition (ASA score 3 or higher, de Jong &amp; Abraham-Insjijn, 1994)</td>
</tr>
<tr>
<td>Poor therapeutic radiation of the head-neck region</td>
</tr>
<tr>
<td>Chronic pain in orofacial system</td>
</tr>
<tr>
<td>Younger than 18 years at time of inclusion</td>
</tr>
<tr>
<td>Reduced mental capacity</td>
</tr>
</tbody>
</table>

Abbreviation(s): ASA, American Society of Anesthesiologists.

### 2.3 | Implant placement and restorative procedures

Implant treatment consisted of the placement of a single implant (Astra Tech Implant System OsseoSpeed TX 3.5x; Dentsply Sirona Implants) in accordance with the manufacturers’ recommendations.

Restorative treatment commenced 3 months later. A screw-retained implant restoration was provided consisting of Resin Nano Ceramic crown (RNC crown, Lava Ultimate, 3M ESPE), bonded extraorally to either a stock (ZirDesign™; Dentsply Sirona Implants) or a CAD/CAM-customized zirconia abutment (Atlantis™; Dentsply Sirona Implants), resulting in a “screwmentation” design of the restorations. Ground material for both abutment types was yttria-stabilized tetragonal zirconium dioxide polycrystal (Y-TZP) according to the requirements of ISO 13356.

After verification of adequate fit and proximal contact points, the abutment fixation screw was tightened, using a wrench at the recommended torque (20 Ncm). The abutment fixation screw was protected by sterile teflon tape, and the screw access hole was sealed with a glass ionomer restorative material (Fuji II; GC Europe). Static occlusion and dynamic occlusion were checked meticulously.

All restorations were made in triplicate: One served as test specimen to be retrieved after 1 year of clinical service (test), one as a pristine control for paired comparison in the planned, future ex vivo experiments (control). A third, geometrically identical set was made to replace the one that was to be retrieved in order to ensure continued function for the patient. The control specimen were carefully stored in an airtight, dry environment until the time of analyses, in order not to alter the characteristics of the material.

All restorations were made by one and the same dental technician. Occasionally, the stock abutment needed to be shortened to obtain adequate occlusal clearance. This was performed under copious water cooling following a transfer key made from silicone. No further preparation or manipulation of the abutments was performed. The abutment type (Figures 1 and 2), stock or CAD/CAM-customized, was randomly allocated to each of the participants using a simple, free online randomization service (www.sealedenvelope.com).

### 2.4 | Raman spectroscopy

Raman spectroscopy is a physical method that can be used to determine the volume percentage of m-ZrO2 in a non-destructive manner (Wulfman et al., 2012; Wulfman, Sadoun, & Lamy de la Chapelle, 2010). It relies on Raman scattering of monochromatic light, usually from a laser source in the visible, near infrared of near ultraviolet range. Its spectrum forms a crystallographic fingerprint of the material studied, among which is m-ZrO2 (Akagawa, Hosokawa, Sato, & Kamayama, 1998). The Raman microscope that was utilized employs a 632.8 nm laser (HeNe Laser, Thorlabs Inc.) and a Shamrock 163 spectrometer with an iDus-420-OE CCD, Andor Technology. An optical Olympus BX51 microscope is used to focus the laser on the region of interest. The scattered light is captured by the same apparatus. The Raman spectrometer was
SCHEPKE ET AL.
calibrated with polystyrene prior to each measuring session, according to protocol.

First, the spectrum of pure, 100% m-ZrO$_2$ was determined, which produces characteristic bands at wavenumbers 384/cm and 477/cm which serve as a fingerprint during further analysis (Figure 3). By adding small amounts of m-ZrO$_2$ to pure tetragonal zirconia powder (Sigma-Aldrich Chemie B.V.), it was determined that these characteristic bands only become discernible at ≥5% volume percentage m-ZrO$_2$, which should be considered the detection threshold of the method.

The abutment samples were stabilized by means of a putty and the mesial, distal, labial, and palatal surfaces of each abutment were examined. The test abutment and its pristine copy were measured consecutively, in random order (Research Randomizer, version 4.0, http://www.randomizer.org), in order to minimize measurement error because of differences in positioning of the specimen. The evaluator was blind to which group the abutment belonged to: test or pristine. The site where most stress was anticipated is located directly below the implant margin at the cervical level (Alsahhaf et al., 2017) and served as the region of interest.

FIGURE 1 Connection area of two abutments with the characteristic appearance after one year of clinical service. The dotted line denotes the different, predominant fracture lines for CAD/CAM and stock implant abutments. CAD/CAM abutment demonstrating a coronal and apical positioned grey line (left, type I). Stock abutment demonstrating a uniformly distributed grey band right (type II).

FIGURE 2 Consort flow diagram. Treatment consisted of implants restored with resin nano-ceramic crowns that were extraorally bonded to (a) ZirconDesignTM (stock) or (b) AtlantisTM (CAD/CAM) customized zirconia abutments.
All data were stored in Andor SOLIS for Imaging X-0000 (version 4.24.30004.0; Andor Technology Ltd.). Subsequently, the data were analyzed to identify the characteristic bands and the volume percentages m-ZrO$_2$ was determined, based on the intensities of the peaks.

### 2.5 | Visual inspection of the connection area and fracture load testing

The restoration (crown–abutment complex) was retrieved from the patients’ mouth, photographed (EOS 600D, EF 100 mm f/2.8 USM Macro and MT-24EX Macro Twin Lite; Canon), and the observations were categorized.

After the Raman analysis, the RNC crowns were carefully removed and the abutments were mounted on a 3.5 mm implant (Astra Tech Implant System OsseoSpeed TX 3.5x; Dentsply Sirona Implants). New fixation screws were tightened to 20 Ncm. Testing of the implant–abutment assemblies was carried out in accordance with the NEN-ISO 14801:2016 standard (International Organisation for Standardisation, 2016) and as described by Truninger et al. (2012), who also tested abutment strength without restorations. In contrast to Truninger, however, the screw access holes were not covered so failure modes could be better established running the fracture test. The implants were embedded in acrylic holders, with the resin at 3 mm from the edge of the implant, simulating some marginal bone loss. Static load was applied at a 45 degree angle to the vertical implant axis. A steel indenter with a round tip (ø 1 mm) was positioned on the highest point of the chamfer outline to have a reproducible point of testing for both samples (aged/ non-aged) and tested in a universal testing machine (MTS 810) at a crosshead speed of 1 mm/min. The specimen were loaded until fracture of the abutment, and maximum load to failure was noted. Evaluation of the maximum load to failure was checked by evaluation of the produced chart where a drop of load could be seen at the point of fracture.

### 2.6 | Failure analysis

Types of fracture were observed using an optical microscope (Wild M3Z; Figure 4). Additionally, in order to observe the fracture

![Figure 3: Raman spectrum for 100% monoclinic zirconia with characteristic, "fingerprint" bands at 384/cm and 477/cm.](image)

![Figure 4: Stock (left) and CAD/CAM-customized (right) zirconia test abutments mounted in the universal test machine. The load cell was place at the highest point of the chamfer outline.](image)
behavior, representative specimen were first sputter-coated with a 3 nm thick layer of gold (80%)/palladium (20%) (90 s, 45 mA; Balzers SCD 030) and analyzed using cold field emission scanning electron microscope (SEM) (LyraTC; Tescan). Fractography analysis was predominantly focused on initiation point and crack propagation (hack lines). By following the hack lines, initiation point was detected and evaluated by means of clinical experience.

2.7 Statistical analysis

The mean fracture load and volume percentages of pristine and clinically aged m-ZrO2 stock and CAD/CAM-customized abutments were calculated, as were relative differences, stratified by abutment type. Verification of normality was tested by means of the Shapiro–Wilk test and eye-balling of the histograms. t Tests for paired samples for each stratum were used. The level of significance was set at $\alpha = 0.05$. All computations were performed using SPSS version 25.0 for Windows (SPSS Inc.).

3 RESULTS

The data for the primary outcome measure (fracture strength) were considered as normally distributed. The mean fracture load values for stock and CAD/CAM-customized zirconia test abutments and their pristine controls, as well as the relative change in strength, are presented in Table 2 and Figure 6. The initial mean fracture strength of pristine CAD/CAM-customized and stock abutments that present with obvious geometric differences was 1.176 N, SD = 74 and 1.237 N, SD = 123, respectively. Clinical function had not noticeably affected the fracture strength of the CAD/CAM-customized abutments. The calculated 95% confidence interval of the difference in effect is (-155.1 N to 67.6 N) (paired sample t test $p = 0.411$). Stock abutments had weakened significantly after 1 year function of their initial average strength. The calculated 95% confidence interval of the difference in effect is (41.9 N to 586.6 N) (paired sample t test, $p = 0.028$).

Failure analysis showed that all included and tested abutments broke inside the implant contour. In contrast to others (Foong et al., 2013), sometimes under high load, the implant had plastically deformed at the neck area. Fracture analysis and SEM analysis showed that the critical flaw for the CAD/CAM abutments was mostly seen just below the neck of the implant and for the stock abutments in the internal connection part, where it was seated. As can be seen in the SEM pictures of one of the stock abutments, the critical flaw was detected at the internal connection and hack lines could be observed after the critical flaw (Figure 7). In three abutment pairs, different failure types were observed and deemed unfit for paired numerical comparison of fracture loads. Sometimes it proved impossible to load the abutment due to a shallow chamfer. Consequently, another 9 pairs of stock and 7 pairs of CAD/CAM abutments were excluded. In total, 11 pairs of stock abutments and 14 pairs of CAD/CAM abutments were included for analysis ($n = 50$ abutments, Figure 2).

Plastic deformation of the titanium test implant after fracture of the abutment was obvious from visual inspection in approximately half of the cases.

On visual inspection of the retrieved abutment samples ($n = 25$ stock and $n = 25$ CAD/CAM abutments), two appearances were predominant: Either two small, separate grey stripes (type I) or a grey band running uniformly along the connection area was noted (type II, Figure 1). Type I was predominantly seen in the CAD/CAM abutments (64%, 16 out of 25), whereas type II was typically observed in the stock implant abutments (96%, 24 out of 25).

![FIGURE 5](image-url) Assembled graph including several representative samples and the anticipated typical spectrum for monoclinic zirconia (m-ZrO2, bottom black line). Note the absence of characteristic bands at 384/cm and 477/cm (which is almost coincident with a band due to tetragonal zirconia) in the samples (colored lines), indicating m-ZrO2 is not present in amounts above the limit of detection of 5% (ww) estimated using model mixtures of monoclinic and tetragonal zirconia.
For Raman spectroscopy, 21 pairs of stock and 23 pairs of CAD/CAM abutments were available for analysis (Figure 2, n = 88 abutments). Volume percentages of m-ZrO$_2$ beyond the detection threshold of 5% were never observed (Figure 3 and Figure 5). Hence, statistical analysis was not performed, and m-ZrO percentages exceeding 25% as set by ISO 13356 (International Organisation for Standardisation, 2015) were not observed for any of the pristine or clinically aged stock or CAD/CAM abutments.

4 | DISCUSSION

Drawing conclusions from in vitro experiments on mechanical behavior and strength of ceramic implant abutments and relating them to clinical practice is troublesome and may yield risky assumptions (Lughi & Sergo, 2010). The test methods that are generally employed differ considerably and their external validity, both with respect to the mechanical as to the hydrothermal accelerated aging methods has not yet been established. This makes time-consuming clinical studies the more relevant, which forms the rationale for the present study.

The volume percentage of monoclinic zirconia (m-ZrO$_2$) and the fracture load of clinically aged stock and CAD/CAM-customized zirconia abutments were pairwise compared to those of pristine exact copies. The fact that these abutments functioned for a period of 1 year under clinical loading is an important asset of this study, although a period of 1 year is still relatively short. Original implants were used for the implant-abutment assembly which also enhances external validity. The use of stainless steel implant analogues, as in some other studies, presumably adversely affects the mechanical behavior under testing conditions (Alsahhaf et al., 2017; Kim, Kim, Brewer, & Monaco, 2009). Although we have found rather large 95% confidence intervals of the difference in effect of the fracture strength of the CAD/CAM-customized abutments, clinical function had not noticeably affected. Stock abutments had weakened significantly after 1-year function of their initial average strength, also within a large 95% confidence interval.

On retrieval and visual inspection of the conical contact area, the two abutment types had quite a different appearance: either a grey band or two grey stripes. Although advanced chemical analysis of the constituents of these grey areas was not performed, it is tentatively suggested that they indicate the contact areas between the zirconia abutment and the titanium implant, resulting from micromovement. Titanium is less wear resistant than zirconia (Klotz et al., 2011; Stimmelmayr et al., 2012). Apicella et al. (2010) evaluated the accuracy of fit of the stock and CAD/CAM abutments of the exact brand used in the present study by evaluating radiographs and ground samples under SEM. From their in vitro study, they concluded that both abutment types exhibited excellent marginal adaptation, without a marked difference between the two. Their findings

**FIGURE 6** Relative change (%) in fracture strength for CAD/CAM-customized and stock zirconia abutments

**FIGURE 7** SEM images of a stock zirconia abutment with an overview (left) and two close-up views from the point of critical flaw (middle and right). The hackles originate from the failure and the fracture moves through the zirconia abutment ending on the other side of the abutment in a symmetrical way. The arrow is pointed at the initiation point of the fracture.
were based on a static setup, without (artificial) loading, whereas our observation was after one year of clinical function suggesting a better, more uniform fit of the stock abutments.

The volume percentage of m-ZrO$_2$ phase and particularly a shift in tetragonal–monoclinic phase distribution is a marker for degradation or aging of the material. Two recent in vitro studies using X-ray diffraction analysis focused on shifts in tetragonal–monoclinic phase transformation after thermocycling and mechanical loading, with contradictory results. Almeida et al. concluded that after a 5-year simulation of clinical use, their zirconia abutments did not show any signs of aging (Almeida et al., 2016). Basilio et al. (2016), however, suggested, based on their findings, that resistance to fracture and the phase stability of implant abutments were susceptible to hydrothermal and mechanical conditions. In these two zirconia studies, different abutment types were used in vitro. In the present ex vivo study, volume percentages beyond 5% were not observed; therefore, substantial degradation of the zirconia material is not expected in 1 year of clinical function. This volume percentage of m-ZrO$_2$ is far below the maximum acceptable percentage of 25% as set by ISO 13356 (International Organisation for Standardisation, 2015).

The original, total study population consisted of 50 patients: 25 stock and 25 CAD/CAM-customized abutments and their pristine copies (Figure 2). Two pairs of stock and CAD/CAM-customized zirconia abutments were used for microstructural characterization using electron backscatter diffraction (EBSD). EBSD mapping is a contemporary, be it destructive technique that allows the detection of a rather small amount of monoclinic phase in nano-crystalline zirconia dental abutments, far smaller than with Raman spectroscopy (for details on EBSD, see Ocelik, Schepke, Rasoul, Cune, & Hosson, 2017). However, the preparation of the samples is rather time-consuming and consequently expensive. The observed volume percentages of m-ZrO$_2$ with EBSD ranged between 0.6%–1.0% and 3.8%–6.2% for CAD/CAM-customized and stock abutments, respectively (Ocelik et al., 2017). These numbers are in line with the data of the other 46 pairs that were analyzed with Raman Spectroscopy in the present study. Others, performing X-ray diffraction measurements on pristine zirconia abutments of another brand also noticed only small residues of monoclinic phase and predominantly tetragonal phase zirconia (Vaquer-Aguilar et al., 2012).

Particularly, the stock abutments exhibited a decrease in fracture strength. The CAD/CAM-customized abutments actually showed a small mean increase in strength, for which no logical explanation can be offered and which may be the result of measurement error. However, Alsahhaf et al. (2017) also noted an increase in fracture load after artificial, dynamic loading in a chewing machine in several groups of their zirconia abutments luted to a titanium base, but also in a group of monolithic YTZ abutments. The stock and CAD/CAM-customized abutments used in the present study are made from the same ground material. The geometry of the abutment varies because the CAD/CAM abutments were individually designed and the stock abutments were not. Furthermore, the internal connection of the stock and CAD/CAM-customized abutments of the studied implant brand is different as well and may play a role. Even though the tapered section of both types of abutments is very similar, the most apical part is not (see also Figure 1). When testing the fracture strength of implant–abutment assemblies, particularly those without a crown, there is a risk that the strength of ceramic abutment wall is tested instead of strength of the implant–abutment connection. The former was considered the principal region of interest and varies per design (see e.g., Apicella, Veltri, Balleri, Apicella, & Ferrari, 2011). For that reason, it was decided to load the abutment at an angle of 45 degrees, instead of 30 degrees as advocated in normative literature (International Organisation for Standardisation, 2016). Both abutment types predominately failed in the internal connection part, but the location was different depending on the abutment type (Figure 1). A slight misfit may have caused a critical flaw. A possible difference in strength resulting from a different design of the transition zone of the individually designed abutments should not have affected the outcome, as we choose to calculate a relative difference in a pairwise setting (i.e., comparing the used and the pristine abutment of the same design).

However, combining the paired test method with a difficult to reach region of interest (i.e., the internal conical connection) had a high cost of many dropouts. A “wrong” fracture location other than the internal connection of one abutment caused the exclusion of the twin sample. Also, we did not make use of a crown–abutment assembly in order to avoid introducing another variable. We used the sole abutment, which caused difficulties in loading the abutments, leading to another high number of dropouts. We believe that this method was suitable to study the strength of the internal connection of a zirconia abutment, which is probably the most vulnerable part, and therefore clinically more relevant than the behavior of the relatively strong transition zone or even the body of the abutment. The dropout could have introduced selection bias, because we could not measure abutments with a shallow chamfer outline. However, the design of the chamfer should not have—to the knowledge of the authors—an influence on the strengths of the abutments.

Unlike Truninger et al. (2012), who compared bending moments of full zirconia abutments to a full titanium control in vitro, both abutment types in the present study showed high fracture strength values of around 1,000 N. Hence, no plastic deformation of the titanium implants was observed in the Truninger study, whereas plastic deformation appeared in approximately half of the samples in the present study. Also, in the present study, a different implant–abutment interface (ETKON abutments on RC implants, Straumann vs. ZirDesign and Atlantis abutments on Astra Tech Implant System OsseoSpeed TX 3.5x; Dentsply Sirona Implants) was used, which makes comparison of the mean fracture strength between studies difficult.

Since fracture resistance is one of the criteria involved in selecting abutment types, a CAD/CAM-customized abutment may be favoured for this particular implant brand. However, the observed fracture load after oblique loading of all abutments, both pristine and clinically aged, is much higher than is to be anticipated in the anterior maxilla as a result of physiological chewing forces that are not likely to exceed 200 N (Regalo et al., 2008; Roldan, Restrepo,
Isaza, Velez, & Buschang, 2016). The suspected difference from visual observation in implant-abutment contact area between the two abutment types was not clinically relevant up to 1 year of functional service.

5 | CONCLUSIONS

After 1 year of clinical service, no difference in fracture strength of for the CAD/CAM-customized zirconia implant abutments with their pristine copies could be demonstrated, whereas the stock zirconia abutments decreased considerably in fracture strength. Maximum values in fracture strength values still exceeded the maximum chewing forces in men.

A substantial degradation of the surface material of zirconia implant abutments was not observed and never exceeded 5%, hence well below the maximum acceptable m-ZrO percentage of 25% as set by ISO 13356 (International Organisation for Standardisation, 2015).

ACKNOWLEDGEMENT

The authors would like to thank prof. Dr. Gerry Raghoebar, Dr. Wim Slot and Dr. Charlotte Jensen for performing the surgical interventions. Furthermore, the authors would also like to thank Dr. Václav Ocelík for his help with the SEM images and fracture analysis and Dr. James Huddleston Slater Jr. for his help in the statistical analysis and interpretation of the data. This study was supported by a grant from Dentsply Sirona Implants, Mõlndal, Sweden, and by the authors’ institutions. Materials were provided free of cost by Dentsply Sirona Implants and 3M.

CONFLICT OF INTEREST

Dr. Schepke and Dr. Cune report grants and other support from Dentsply Sirona Implants, Mõlndal, Sweden, and non-financial support from 3M during the conduct of the study. Dr. Browne, Dr. Abdolahzadeh, Mr. Nijkamp and Dr. Gresnigt have nothing to disclose.

AUTHOR CONTRIBUTIONS

US built the design of the study and collected the data. Funding was secured by MSC. He designed the study together with US, revised the article critically and added most of the content. WRB, SA, and JN substantially contributed to the Raman spectroscopic analysis, revised the article critically, and added content. MMMG performed the fracture load experiment, performed the SEM analysis, revised the article critically, and added content.

ORCID

Ulf Schepke  https://orcid.org/0000-0002-5995-0735

REFERENCES


**SUPPORTING INFORMATION**

Additional supporting information may be found online in the Supporting Information section at the end of the article.

**How to cite this article:** Schepke U, Gresnigt MMM, Browne WR, Abdolahzadeh S, Nijkamp J, Cune MS. Phase transformation and fracture load of stock and CAD/CAM-customized zirconia abutments after 1 year of clinical function. *Clin Oral Impl Res*. 2019;00:1–11. https://doi.org/10.1111/clr.13442