Effect of Aging on the Load-to-Failure of Molar Implant-Supported Monolithic Zirconia Crowns: A Laboratory Study

Varun Garg, BDS, DCD

Specialist prosthodontist, Private Practice, Melbourne, Australia.

Roy B. Judge, BDS, LDS, RCS, MDSc, PhD

Associate Professor, Head of Prosthodontics, Melbourne Dental School, Melbourne University, Melbourne, Australia.

Jaafar Abduo, BDS, DClinDent, MRACDS (Pros), PhD

Associate Professor in Prosthodontics, Convenor of Postgraduate Diploma in Clinical Dentistry (Implants), Melbourne Dental School, Melbourne University, Melbourne, Australia.

Joseph E. A. Palamara, BDSc, Dip Ed, PhD

Associate Professor, Melbourne Dental School, Melbourne University, Melbourne, Australia.

Corresponding author:

Associate Professor Jaafar Abduo

Restorative Section, Melbourne Dental School, Melbourne University

720 Swanston Street, Melbourne, Victoria, 3000, Australia

Email: jaafar.abduo@unimelb.edu.au

Phone: +61 3 9035 8998

Fax: +61 3 9341 1599

Submitted November 11, 2019; accepted June 10, 2020
**Purpose:** To evaluate the effect of hydrothermal aging on the load to failure and number of cycles to failure of implant-supported monolithic zirconia molar crowns under cyclic loading. **Materials and Methods:** Twenty identical implant-supported monolithic zirconia crowns with molar morphology were produced. Half of the crowns were aged according to ISO standard 13356 to simulate 5 years in vivo. The non-aged crowns served as a control group. All crowns were subjected to cyclic loading with increasing increments of load until failure. The load to failure, the number of cycles to failure, and the failure pattern were determined for each crown. **Results:** The load to failure values were 3,630 N (SD: 547.8 N) and 3,640 N (SD: 389.3 N) for the non-aged and aged crowns, respectively. The non-aged crowns failed after 33,480.1 cycles (SD: 23,138.4 cycles), and the aged crowns failed after 28,456.1 cycles (SD: 10,158.7 cycles). There was no significant difference between the two groups for the load to failure or number of cycles to failure. The predominant form of failure was catastrophic crown fracture, which was observed for all the non-aged crowns and 9 of the aged crowns. **Conclusion:** Within the limitations of this study, aging of the implant-supported monolithic zirconia crowns with molar morphology did not affect the load to failure or the number of cycles to failure under cyclic loading. Since all the crowns failed at much higher loads than the expected physiologic loads, clinical application of implant-supported monolithic zirconia crowns to replace missing molars seems reasonable. *Int J Prosthodont* 2021. doi: 10.11607/ijp.6815

Recently, there has been a shift in the restoration methodologies of single implants that is driven by advanced technologies and the development of high-strength ceramic materials. Zirconia has the advantages of being biocompatible, inert, durable and esthetic. The most commonly applied form of zirconia in dentistry is the polycrystalline stabilized zirconia. The superior mechanical properties of stabilized zirconia are attributed to transformation toughening, a feature that resists crack propagation by a localized slight increase in the size of zirconia crystals at the crack site.\(^1,2\)

Despite the promising outcome of zirconia restorations, a few limitations were identified, such as the high rate of ceramic veneer chipping and fracturing of zirconia infrastructure.\(^3,4\) To overcome these problems, recommendations were made to design a full-contour monolithic zirconia prosthesis, where the whole prosthesis is composed solely of zirconia.\(^5-7\) With this approach, a thicker zirconia infrastructure is used. However, as a result of the monolithic zirconia design, the zirconia material is exposed directly to the oral environment. The temperature and the fluids in the oral cavity will subject the material to hydrothermal aging, where the crystals at the exposed surface transform from the tetragonal phase to the monoclinic phase,\(^1,8,10\) which is more susceptible to
microcrack formation, water penetration, crystal pull-out and progressive surface deterioration. Thus, it is anticipated that the frequency of failure of zirconia restorations is accentuated by hydrothermal aging. Monolithic zirconia application has been shown by several clinical studies to be promising on posterior natural teeth. However, the clinical studies on implant-supported monolithic zirconia restorations are limited, and cannot yet be used to generate clinical recommendations. Further, the monolithic zirconia restorations on implants are subjected to a more challenging mechanical environment than natural teeth. This is attributed to the lack of periodontal ligament, presence of thin sections of zirconia near the implant connection, and the presence of screw access within the implant-supported crown. Therefore, in an attempt to understand the behavior of implant-supported monolithic zirconia crowns with natural anatomy under a simulated laboratory environment, the aim of this study was to evaluate the effect of hydrothermal aging on the load-to-failure and number of cycles to failure of implant-supported monolithic zirconia crowns. The null hypothesis is that there is no difference in the load-to-failure and the number of cycles to failure of implant-supported monolithic zirconia crowns, without and with simulated hydrothermal aging.

**MATERIALS AND METHODS**

**Implant-Supported Crowns Preparation**

The specimens of the study were single implant-supported monolithic zirconia crowns fabricated by a commercial manufacturer (NobelProcera; Nobel Biocare AB, Zürich-Flughafen, Switzerland). The crowns were planned to fit deep conical connection implants with a 5 mm diameter and 10 mm length (NobelActive, Nobel Biocare, Göteborg, Sweden). According to the manufacturer’s recommended protocol, the crowns were attached to prefabricated intermediate titanium inserts. The titanium inserts were cementless and connected to the zirconia crowns by friction lock (NobelProcera FCZ Implant Crown; Nobel Biocare, Göteborg, Sweden). The head of the retaining screw seats on the zirconia interface. As a result, this design allowed for the attaching of the zirconia crown on the titanium insert without cementation. The crown design followed the shape of a standard maxillary first molar and was completed using a CAD design software (DTX Studio, Nobel Biocare, Zurich, Switzerland). The dimensions of the crown were 7.5 mm in height, 10.4 mm mesiodistal length, 11.5 mm bucco-lingual width and 10.7 mm bucco-lingual cervical width. The manufacturing procedure involved milling the zirconia crowns at the pre-sintered state. This was followed by sintering and mechanical polishing by the manufacturer to clinically acceptable standards for the insertion of the crowns. After receiving the crowns from the manufacturer, none of them were polished or adjusted.
The ageing protocol followed the ISO standard 13356, which recommends exposing polycrystalline zirconia to 134°C for 5 hours in a steam autoclave (W&H Sterilizers, Lisa 522, Prion B 134 cycle, Bürmoos, Austria) at 0.2 MPa in order to induce accelerated hydrothermal aging. This protocol was found to simulate zirconia aging equivalent to at least 5 years in vivo. In this study, only the zirconia components of the specimens were subjected to aging.

The implant of each specimen was affixed in the middle of a polyvinyl mould with a diameter of 25 mm and height of 18 mm. The mould was subsequently filled with autopolymerizing acrylic resin (Unifast Triad III; GC Corp, Tokyo, Japan) up to the height of the implant collar to prevent any contact between the resin and the crown. To ensure reproducible implant positioning, a dental surveyor arm connected to an impression coping was used to place the implants. A period of at least 24 hours was allowed for the resin to polymerize completely. The zirconia crown with the intermediate titanium insert was attached to the implant by a titanium clinical screw that was torqued to 35 Ncm. The screw access hole was sealed by polytetrafluoroethylene tape followed by increments of composite resin (Reflectys, Nano-Hybrid Composite, Paris, France) (Fig 1). Each increment was light cured for 20 seconds followed by a final light cure for 40 seconds.

Prior to the main experiment, a pilot study was conducted on 3 non-aged and 3 aged specimens. These specimens were loaded by single static load-to-failure. The non-aged specimens failed at an average load of 7330.6 N, while the aged specimens failed at an average load of 5324.0 N. The results of the pilot study were used to calculate the required sample size by a statistical software program (Sample Power 20; IBM Corp, Armonk, New York, USA). Assuming 80% statistical power and a 5% significance level, at least 8 specimens were required for every group. For the main study, a total of 20 identical crowns were produced. The crowns were equally and randomly divided into 2 groups: non-aged and aged. The non-aged crowns served as the control group.

**Cyclic Loading**

The crowns were tested under similar cyclic loading conditions to simulate masticatory forces. The specimens were held in position with a customized brass jig on a closed loop servohydraulics machine (MTS 810 Materials Test System; MTS System Corp; Eden Prairie, Minnesota, USA). The direction of the applied load was parallel to the long axis of the implant to simulate the expected forces on a molar implant. A stainless-steel ball with a diameter of 8 mm was attached at the end of the loading platen to represent masticatory forces applied by an opposing cusp. This diameter was selected to ensure the ball contacts the cuspal inclines without seating on the screw access. The centre of the ball was positioned at 1 mm mesial to the centre of the specimen, which corresponded to the mesial fossa. As a result, the ball applicator contacted three cuspal inclines of the molar crown.
during loading. A new ball was used for every specimen. A similar location of the ball applicator was ensured on each crown with the aid of a magnifier.

During testing, all specimens were kept moist with saline soaked gauze. A stepped cyclic loading protocol was used to simulate masticatory forces for all the crowns. According to the values obtained from the pilot study, the loading started at an initial force of 3000 N for 20,000 cycle. This was followed by incremental increases to 3500 N, 4000 N, and 4400 N. For each increment, a maximum of 20,000 cycles were applied. The crowns of the 2 groups were randomly tested. An interlock was set within the servohydraulics machine to detect any sudden increase in displacement such as fracture of the crown or any component of the specimen. For each crown, the maximum applied load-to-failure during cyclic loading, and the total number of cycles endured were recorded. After failure of each crown, the mode of failure was determined by visually inspecting the crown under a stereomicroscope (Leica S8APO; Leica Microsystems GmbH, Wetzlar, Germany) and a scanning electron microscope (FEI Quanta FEG Esem, Hillsboro, Oregon, USA), to further identify the origin of the fracture. The expected failure patterns were one or a combination of catastrophic crown fracture, cervical crown fracture, titanium insert deformation/fracture, screw deformation/fracture, and implant deformation/fracture.

Statistics

The means, standard deviations, medians, maximum values and minimum values were calculated for the load-to-failure and number of cycles to failure for each group. The presence of a statistical difference between the 2 groups was evaluated using the Mann-Whitney U test through a statistics software (SPSS for Windows, v23; SPSS Inc, Chicago, Illinois). The level of significance was set at 0.05.

RESULTS

The results of the study were presented in Table 1. The crowns of the 2 groups failed at similar load-to-failure values \( (P = .96) \) (Fig 2a). The non-aged crowns failed at a mean load-to-failure of 3630 N \( (SD = 547.8 \text{ N}) \), and the aged crowns failed at a mean load-to-failure of 3640 N \( (SD = 389.3 \text{ N}) \). In relation to the total number of cycles before failure, the non-aged crowns failed after an average of 33480.1 cycles \( (SD = 23138.4 \text{ cycles}) \) and the aged crowns failed at lower average cycles \( (\text{mean} = 28456.1 \text{ cycles}; \ SD = 10158.7 \text{ cycles}) \). There was no statistically significant difference between the 2 groups \( (P = .54) \) (Fig 2b).

The predominant pattern of failure was catastrophic crown fracture through the middle of the crown that involved the screw access (Fig 3a). This had occurred for all the non-aged crowns and 9 aged crowns (Table 2). The fractures appeared to initiate from the point of loading, where the ball
contacted the cuspal incline and moved apically, rather than initiating from the screw access (Fig 3b). The remaining aged crown failed by fracture at the cervical region, where a small chip was noted in the cervical area of the crown surrounding the titanium insert. Two titanium inserts of the non-aged group, and 1 titanium insert of the aged group showed signs of plastic deformation. One retaining screw of the aged group was deformed after loading. None of the metal components fractured after loading, and none of the implants showed any sign of fracture or deformation.

**DISCUSSION**

According to the present study, zirconia aging had no effect on the load-to-failure of implant-supported monolithic zirconia molar crowns when subjected to a stepped cyclic loading. Therefore, the hypothesis that there is no difference in the load-to-failure and the number of cycles to failure of implant-supported monolithic zirconia crowns, without and with simulated aging, was accepted. All crowns fractured at high loads that started at 3000 N, which is much higher than the average reported bite force in the molar region (630 N), and even higher than the extreme bite force occasionally reported on posterior implants (1200 N). Therefore, it can be speculated that this restoration modality is suitable for restoring posterior implants. In the present study, a stepped cyclic loading was applied, since it is a more realistic laboratory simulation of the clinical environment than single static load-to-failure. It is anticipated that the loading mechanism influences the failure pattern of implant-supported crowns, which are composed of multiple components exhibiting inevitable micromovements. This may explain the generally higher load-to-failure values obtained in the pilot study compared with the values of the main experiment.

Several studies on thin zirconia bar specimens reported that the depth of phase transformation of exposed zirconia increased with simulated aging time, and the increased amount of the monoclinic phase decreased the flexural strength of zirconia. However, the present study did not support such findings, and the simulated aging of 5 years did not seem to affect the load-to-failure of monolithic zirconia crowns on implants. This can be due to the dimension and bulkiness of the crowns, as opposed to the thin bar specimens of 0.2 mm thickness used by the earlier studies. Moreover, it can be speculated that the magnitude of surface transformation after the simulated aging period was not sufficient to induce weakening of the implant-supported crowns. In accordance with this study, other laboratory investigations of large zirconia specimens found a minimal impact of aging. For example, using 1.5 mm disk specimens, Camposilvan et al showed that the aging-induced phase transformation occurred only at the 2-6 µm superficial layer, which had no effect on biaxial strength. In addition, Alsahhaf et al reported that for anterior implants, aging had a minimal influence on the mechanical performance of zirconia abutments on titanium inserts. Likewise,
Spies et al evaluated the effect of aging on zirconia implants, and they found that the monoclinic layer thickness increased by 2-3 µm when exposed to water. However, this monoclinic layer had a minimal effect on fracture resistance and surface roughness. Therefore, although phase transformation on the surface of zirconia specimens can still be observed, if the proportion of the transformation to the rest of the specimen is minimal, it may not be sufficient to cause actual weakening of the zirconia crowns. Thus, stronger evidence on the effect of aging on the mechanical performance of zirconia prostheses is yet to be provided.

The full-contour monolithic design of the zirconia crowns appeared to be beneficial to increase the load-to-failure of zirconia crowns. This can be due to implementation of maximum material bulk for the fully contoured crowns. In this study, the advantage of the monolithic zirconia design was evident since the predominant mode of failure was bulk fracture of the crowns, where the cracks initiated from the point of contact between the crown and the loading ball. Interestingly, the cracks propagated apically through the thickest zirconia sections, rather than initiating from the screw access or the apical aspect near the implant connection (Fig 3). In addition, the use of titanium inserts eliminates the drawback of the direct seating of the zirconia abutment interface on the implant, where the screw head is seated directly on thin section of zirconia. Further, monolithic design and fabrication reduces the likelihood of adjusting the restoration, which accentuates the phase transformation, the development of microcracks, and the reduction of fracture resistance.

For posterior implants, a recent in-vitro study evaluated the load-to-failure of monolithic high translucent screw-retained zirconia crowns under static loading. The study showed similar load-to-failure values to this study (3440 N). The authors concluded that the implant-supported monolithic zirconia crowns exhibited significantly higher load-to-failure when compared to other tested groups, such as layered high- and low-translucent zirconia restorations and conventional metal ceramic restorations. Likewise, Preis et al and Elsayed et al showed that implant-supported monolithic zirconia crowns failed at similarly high loads (4817-5529 N). Today, with the advancement of digital technologies in the form of virtual restoration design and accurate fabrication techniques, it is feasible to quantitatively measure and avoid critically thin zirconia sections, which may further optimize the clinical outcome.

While this study clearly indicated no effect of aging on the load-to-failure and the number of cycles to failure, it is important to emphasize that it suffers from some limitations related to the simulation of clinical conditions. In this study, only axial loading of the specimens was used, which may have resulted in high load-to-failure of the implant-supported crowns. Dynamic loading and oblique forces may subject the crown to more tensile stresses and cause different failure patterns, such as screw loosening or wear of the titanium insert. The aging process followed in this study...
may not reflect the complex thermomechanical aging process that can take place intraorally. None of the specimens of the present study were adjusted, which may not reflect clinical procedures where minor surface adjustments are necessary. Such procedures may further contribute to the aging degradation and cause additional mechanical deterioration.2

CONCLUSIONS

Within the limitations of the present study, it can be concluded that aging of the implant-supported monolithic zirconia crowns did not affect the load-to-failure and the number of cycles to failure, as the aged and non-aged groups failed at similar loads and patterns. All the crowns failed at a much higher loads than the expected physiological loads.

ACKNOWLEDGMENTS

The authors acknowledge the contribution of Nobel Biocare, Kloten, Switzerland in providing the implant components of the study (2015-1360). The study was funded by the Australian Prosthodontic Society and the Melbourne Dental School Research Higher Degree Funding. The authors would like to thank Mr Geoffrey Adams for his statistical support and Mr George Thalassinos for his technical support.

REFERENCES

List of Figures

Fig 1  The final assembly of the implant zirconia monolithic crown attached on the implant prior to loading.
Fig 2  Box-and-whisker plots of the results of the study for the non-aged and aged crowns. (a) Load-to-failure (N). (b) Number of cycles to failure.
Fig 3 Examples of catastrophic failure of the zirconia crowns. (a) An image illustrating crown fracture involving the screw access. (b) A scanning electron microscope image of a crown fracture demonstrating the crack initiation under the point of loading (black arrow) followed by apical crack propagation (white arrows).
List of Tables

**Table 1** Mean, standard deviation, median, maximum value and minimum value of the load-to-failure and number of cycles for the non-aged and aged crowns

<table>
<thead>
<tr>
<th></th>
<th>Group</th>
<th>Mean</th>
<th>Standard deviation</th>
<th>Median</th>
<th>Maximum</th>
<th>Minimum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load-to-failure (N)</td>
<td>Non-aged</td>
<td>3630.0</td>
<td>547.8</td>
<td>3500.0</td>
<td>4400.0</td>
<td>3000.0</td>
</tr>
<tr>
<td></td>
<td>Aged</td>
<td>3640.0</td>
<td>389.3</td>
<td>3500.0</td>
<td>4400.0</td>
<td>3000.0</td>
</tr>
<tr>
<td>Number of cycles</td>
<td>Non-aged</td>
<td>33480.1</td>
<td>23138.4</td>
<td>37951.5</td>
<td>65351.0</td>
<td>3492.0</td>
</tr>
<tr>
<td></td>
<td>Aged</td>
<td>28456.1</td>
<td>10158.7</td>
<td>26778.0</td>
<td>40800.0</td>
<td>10739.0</td>
</tr>
</tbody>
</table>

**Table 2** The mode of failure of the non-aged and aged crowns

<table>
<thead>
<tr>
<th></th>
<th>Group</th>
<th>Mode of failure</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Catastrophic crown fracture</td>
</tr>
<tr>
<td>Load-to-failure (N)</td>
<td>Non-aged</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Aged</td>
<td>9</td>
</tr>
</tbody>
</table>