Influence of dental tissue substructure and dimension on the fracture strength of lithium disilicate and zirconia ceramics

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Abstract

**Purpose:** To assess the influence of substructure and dimension on the fracture strength of ceramic discs made from both lithium disilicate ceramic and zirconia. **Materials and Methods:** A total of 128 intact maxillary third molars were collected, and standardized enamel and dentin discs were fabricated. Lithium disilicate ceramic (IPS e.max CAD, Ivoclar Vivadent; n = 64) and zirconia (Katana, Kuraray; n = 64) discs with 0.5-mm (n = 32 IPS and n = 32 Katana) and 1-mm (n = 32 IPS and n = 32 Katana) thickness were produced, and each group was divided into two subgroups (n = 16 each) that were luted to the enamel or dentin discs using Panavia V5 (Kuraray). Half of the specimens in each subgroup were aged (chewing simulation and thermocycling), and all specimens were loaded until fracture in a universal testing machine (Z010, Zwick/Roell). Differences between Katana and IPS with respect to enamel and dentin as substructure and the thickness of the ceramic were analyzed by use of a nonparametric test (Mann-Whitney *U* test). **Results:** In un-aged specimens, fracture loads were not significantly (*P* > .05) different between zirconia and IPS specimens for 1- or 0.5-mm thickness. However, fracture loads were significantly higher (*P* < .001) in specimens supported by enamel, independent of the ceramic material. In aged specimens, the fracture loads of all specimens were significantly (*P* < .01) higher when supported by enamel; however, in the 0.5-mm groups, zirconia achieved significantly higher breaking loads than IPS when luted to dentin. **Conclusion:** When 0.5-mm ceramic discs were luted to dentin, zirconia outperformed IPS with respect to breaking loads. *Int J Prosthodont* 2021. doi: 10.11607/ijp.7451

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1. Introduction

In the last decades, all ceramic restorations succeeded in the dental market. Although ceramic is a brittle material, zirconia monolithic restorations overcame the disadvantages of chipping and fracture. This development was promoted by the considerably increasing use of computer aided design and manufacturing techniques. These techniques enable the application of innovative materials using time- and cost-efficient digital workflows. Numerous companies offer milling machines and an increasing number of machinable materials are available to cover almost all requirements of up-to-date fixed dental restorations: all ceramic FDPs, crowns, partial crowns etc. can be designed and produced, both lab-side or chair-side.

Tooth wear is an increasing problem in both older subjects \(^1\) and adolescents, \(^2\) being a result of abrasion, attrition and erosion.

Although prevalence data which can be found in literature are heterogeneous, ranging from 0%-54% \(^1\) or 20%-45% \(^3\) in permanent dentition, the need for dental restorations in selected patients is undoubtable.\(^4\)

Occlusal veneers are a contemporary restorative approach indicated for teeth with occlusal wear. Several materials are available to replace the worn occlusal interface using occlusal veneers: nanocomposites, polymer-infiltrated ceramics, zirconia, lithium disilicate reinforced glass-ceramics, zirconia-reinforced lithium silicate glass-ceramics, leucite reinforced glass-ceramics or cross-linked polymethyl methacrylate (PMMA) material. Ceramic materials have some persuasive advantages compared with nanocomposites, PMMA and polymer-infiltrated ceramics: biocompatibility, \(^5\) esthetics, \(^5\) and reduced wear. \(^6\)

However, the brittleness of traditional feldspathic ceramics requires a thickness of at least 2mm for occlusal veneers,\(^7\) which is sometimes hard to achieve. Thus, the use of zirconia, which is less brittle than feldspathic ceramics, might be beneficial. In a clinical setting,
several studies showed a good clinical performance\textsuperscript{8, 9} of both zirconia and lithium disilicate restorations in full-coverage restorations

However, when restoring teeth using occlusal veneers, a special issue with respect to this kind of restoration has to be taken into consideration: the influence of the substructure (dentin, composite or enamel) on fracture resistance of the occlusal veneer.\textsuperscript{10-12} The mechanical properties of enamel and dentine are substantially different\textsuperscript{13}: the range of the elastic modulus of enamel is between 60 and 130 GPa and that of dentine between 11 and 45 GPa. The large variation within both enamel and dentine can be explained by different testing methods and heterogeneous mechanical properties within both enamel and dentin. E.g. for enamel the elastic modulus decreases gradually from the surface to the dentine-enamel-junction, and the elastic modulus of dentine decreases slowly towards the pulp cavity.\textsuperscript{13} Consequently, the fracture strength seems to be higher when a veneer is luted to superficial enamel instead of deeper parts of dentine.\textsuperscript{11} This might have an influence on the survival of occlusal veneers, especially when the occlusal veneers are luted to dentine in severely worn teeth. In these cases, the use of ceramics presenting a high fracture resistance might be useful\textsuperscript{14} to compensate for the reduced elastic modulus of dentine as brittle materials need to be supported by the substrate below in combination with a good adhesive interface. If the luting, the design, and the environment are optimal, an unibody design restoration is formed.\textsuperscript{15} Although some studies\textsuperscript{12, 16} have assessed the influence of the substructure (dentine versus enamel) on the fracture strength of ceramic materials, in the present study the mechanical properties of two ceramic materials were compared – in an attempt to identify the most suited material - and the specimens have been prepared using an innovative approach\textsuperscript{17} resulting in highly standardized specimens. The null hypothesis of the present in-vitro study was that the substructure (enamel/dentine) does not significantly influence fracture strength in both zirconia and lithium disilicate ceramics when used for occlusal veneers.
2. Material and methods

The study protocol conformed to the principles outlined in the German Ethics Committee statement for the use of human body material in medical research (Central German Ethics Committee Statement: The use of human body materials for the purpose of medical research [in German];2003) and was approved by the local review board (#15/15).

2.1 Tooth preparation

128 intact upper third molars were collected and stored in 1% chloramine-T solution (storage time between 2 and 10 weeks). Afterwards, the root surfaces were cleaned by use of scalers and isopropyl alcohol. All the teeth were prepared with a standardized and tested method with 3D printed devices for the production of enamel-dentine and dentine tooth discs. This was done in order to get on the one hand specimens covered with enamel and on the other specimens without enamel (Fig. 1 a-c). All specimens were milled perpendicular to the dentinal tubules and had a diameter of 5mm. Finally, the thickness of both enamel and dentine was controlled using a microscope (AXIO Lab.A1, Carl Zeiss AG, Oberkochen, Germany).

2.2 Preparation of the ceramic specimens

Lithium disilicate (IPS e.max CAD, Ivoclar, VITA, Germany; LOT X57291) specimens with a thickness of 1mm (n=32) and 0.5 mm (n=32) were designed with Autodesk Inventor 2020 (Autodesk, San Rafael, USA) and milled (MC XL, Sirona, Bensheim, Germany). The used software for milling was CEREC SW Version 5.1.1 (Sirona, Bensheim, Germany). Additionally, zirconia (Katana Zirconia Block STML, Kuraray Noritake Dental, Japan; LOT DVCUK) specimens were milled according to the Lithium disilicate specimens (MC XL,
Sirona, Bensheim, Germany) with a thickness of 1mm (n=32) and 0.5 mm (n=32) using the same .stl-file of the lithium disilicate specimens.

Thus, the following groups were established:

- 1. 0.5mm enamel, 2.5mm dentine and 1 mm Katana (n=16)
- 2. 0.5mm enamel, 2.5mm dentine and 1 mm IPS e.max (n=16)
- 3. 3mm dentine without enamel and 1mm Katana (n=16)
- 4. 3mm dentine without enamel and 1mm IPS e.max (n=16)
- 5. 1mm enamel, 2.5mm dentin and 0.5mm Katana (n=16)
- 6. 1mm enamel, 2.5mm dentin and 0.5 mm IPS e.max (n=16)
- 7. 3.5mm dentine without enamel and 0.5mm Katana (n=16)
- 8. 3.5mm dentine without enamel and 0.5mm IPS e.max (n=16)

These different groups should simulate different degrees of tooth wear: when excessive tooth wear is present, thicker ceramic specimens have to be used, predominantly luted to dentine. If wear is moderate, thinner restorations have to be used, predominantly luted to enamel.

2.3 Luting ceramic discs to tooth specimens

2.3.1 Lithium disilicate

The surface of the tooth specimens was conditioned using Panavia V5 tooth primer for 20 seconds (Kuraray Noritake Dental, Japan) and air-dried using a syringe. The surface of the lithium disilicate specimens (n=64) was etched with 5% hydrofluoric acid (Kuraray Noritake Dental, Japan) for 20 seconds, rinsed with water (60 seconds) and dried with air using a syringe. Subsequently, a thin layer of Clearfil Ceramic Primer Plus (Kuraray Noritake Dental, Japan) was applied and dried. Finally, Panavia V5 (Kuraray Noritake Dental, Japan) was applied on the ceramic specimen and the ceramic and tooth specimen were put together with 10N in a 3D printed cementation device (Fig. 2a), excess was removed, and the tooth-
ceramic complex was light-cured for 30 seconds. This procedure was performed for all specimens in the same, standardized way.

2.3.2 Zirconia

The surface of the tooth specimens was conditioned using Panavia V5 tooth primer for 20 seconds (Kuraray Noritake Dental, Japan) and air-dried using a syringe.

The surface of the zirconia specimens (n=64) was air abraded using alumina power (P-G400, Harnisch & Rieth, Winterbach, Germany, aluminium oxide, 50 µm, 1.0 bar, distance 10 to 12mm) and cleaned for 2 minutes with 95% isopropyl alcohol in an ultrasonic device (Sonorex super RK 102H; Bandelin, Berlin, Germany). Subsequently, a thin layer of Clearfil Ceramic Primer (Kuraray Noritake Dental, Japan) was applied and dried. Finally, Panavia V5 (Kuraray Noritake Dental, Japan) was applied on the ceramic specimen and the ceramic and tooth specimens were put together with 10N in a 3D printed cementation device (Fig. 2a), excess was removed, and the tooth-ceramic complex was light-cured for 30 seconds.

After cementation, all specimens were stored at 36±2°C for 24 hours. Finally, the specimens were embedded with Technovit 7072 (Kulzer GmbH, Hanau, Germany) in specimen holders. The specimens were placed in a 0.3mm deep mold. The diameter was with 5mm the same as the specimen (Fig. 2b).

2.4 Chewing simulation and thermocycling

Half of the specimens (n=64) were exposed to 5000 cycles from 5°C to 55°C in a thermal custom-made cycling machine (dwell time 30s, intermediate pause 7s). The specimens were fixed in a 3D printed holder (Fig. 2c). This was done before the embedding in the specimen holders. In this device up to 80 specimens can be thermocycled together. The specimens were placed at equal distances and the contact to the specimen holder was reduced to a
minimum. The contact area to the holder was approximately 20% of the disc. The water can surround nearly the whole plate during cycles. This was done to guarantee a constant heat exchange during the artificial ageing. The specimens then underwent mechanical artificial ageing by use of a chewing simulator (chewing simulator CS-4, SD Mechatronics GmbH, Germany, 1.0 million cycles, purely vertical movement with mass m=5kg, descending speed v=90mm/s, 1.2Hz loading frequency). The total of 1.0 million cycles in the chewing simulator and 5000 thermocycles were completed to simulate approximately four years of clinical use.\textsuperscript{18-20} The other half (n=64) was not exposed to artificial ageing.

2.5 Ultimate loading

All specimens were loaded until fracture in a universal testing machine (Z010, Zwick/Roell, Germany) with a crosshead speed of 1mm/min. The load was applied with an indenter at the centre of the specimen, perpendicular to the axis of the specimen, until fracture. The indenter was a 6mm ball sphere embedded into a holder from the chewing simulator (Fig. 2b).

2.6 Statistical analysis

Descriptive statistical analysis (median, interquartile range) was conducted. The Kolmogorov-Smirnov test was used to test for normal distribution of the data. Differences between Katana and IPS e.max with respect to enamel and dentine as substructure and thickness of the ceramic were analysed by use of a non-parametric test (Mann-Whitney U-Test) including the Bonferroni correction. All statistical analyses were performed by use of SPSS (SPSS Statistics 25, IBM Chicago, Illinois 60606, USA).

3. Results

All specimens which failed at artificial aging in the chewing simulator were set to 50N. This is the force of every cycle of the chewing simulator. During ageing, two 0.5 mm zirconia and
five 0.5 mm lithium disilicate ceramic specimens failed in the groups without enamel support. Additionally, one specimen failed in the 1 mm lithium disilicate ceramic group without enamel. Thus, all 120 specimens could be tested in the universal testing machine to obtain breaking loads. The Kolmogorov-Smirnov test showed that not all the data were normally distributed. Consequently, non-parametric tests were used (Mann-Whitney U-Test). In the subsequent pairwise comparison using the Mann-Whitney U-test, the previously selected level of significance (p=0.05) was adjusted to p=0.0063 by the Bonferroni correction.

3.1 Initial breaking loads (n=64)
In un-aged specimens, the fracture loads were not significantly (Mann-Whitney U-Test; p>0.05) different between zirconia and lithium disilicate ceramic specimens for 1 and 0.5mm thickness. However, the fracture loads were significantly higher (p<0.001) in specimens supported by 1 and 0.5mm enamel compared to specimens supported by dentine, independent of the ceramic material which was used. The fracture loads are given in table 1, 2 and in figure 3 (Whiskers-Boxplots diagram).

3.2 Fracture loads after artificial ageing (n=56)
In general, the fracture loads were significantly (Mann-Whitney U-Test; p<0.01) higher when the ceramic specimens were supported by enamel instead of dentine except for 0.5mm lithium disilicate ceramic (p=0.015). This result was found for both zirconia and lithium disilicate ceramics. In 1mm thick ceramic specimens luted to 0.5mm enamel and 2.5mm dentine, zirconia and lithium disilicate ceramic did not show significant differences in fracture load (Mann-Whitney U-Test; p=0.06), although a clear trend for better results in zirconia specimens was found. The same result was found for 0.5mm thick ceramic specimens which were luted to 3.5mm dentine (Mann-Whitney U-Test; p=0.44). However, two thin (0.5 mm) occlusal veneers of the zirconia group and five of the lithium disilicate group luted to dentine
failed during ageing. Some of the remaining specimens achieved breaking loads below 350 N. For the other specimens (Fig. 4: group 2 and group 3) with 1mm thick specimens luted to 3mm dentine and 0.5mm thick specimens luted to 1mm enamel and 2.5 dentine, zirconia and lithium disilicate showed no significant differences (Mann-Whitney U-Test; p=0.01 and p=0.04, respectively). The fracture loads of the aged specimens are given in table 1, 2 and in figure 4 (Whiskers-Boxplots diagram), showing that zirconia performed better in all groups, although the level of significance was reached for two groups only.

4. Discussion

In the present study, the null hypothesis had to be rejected: ceramic discs luted to enamel achieved significantly higher fracture loads compared to ceramic discs luted to dentine. This result was found for both aged and un-aged specimens. However, in aged specimens, 1 mm thick zirconia discs outperformed lithium disilicate discs with respect to fracture loads when dentine was the substructure, whereas in un-aged specimens this difference could not be found. Additionally, in thin ceramic discs (0.5 mm), zirconia showed significantly higher fracture loads when the substructure was enamel. The results for 0.5 mm ceramic discs luted to dentine were devastating, as in both groups the ceramic discs failed during ageing and some of the remaining fracture loads were below 350 N. When the ceramic discs are luted to enamel, the fracture loads are above 930 N for zirconia and 700 N for lithium disilicate ceramic after ageing. Although maximum biting forces in the natural dentition can possibly range between 289 ± 103 N and 738 N in the premolar and molar regions,21, 22 forces during function are lower, because these forces are non-axial.23 Thus, thin ceramic discs might withstand even maximum biting forces in the molar region clinically, whereas lithium disilicate ceramic discs might withstand maximal biting forces in the premolar region, only. However, both materials might withstand physiological forces during function. In the present study it was shown that both zirconia and lithium disilicate ceramic were affected by artificial
ageing with respect to load-bearing capacity. This effect might base on the weakening of the adhesive joints during artificial ageing/water storage. However, although the adhesive interface in etchable ceramics is influenced by hydrolysis, too, the micromechanical retention compensates the effect of the chemical degradation.

Two other studies assessed the influence of substructure (enamel/dentine) on the load-bearing capacity of ultra-thin occlusal veneers. In these studies, the breaking loads of lithium disilicate ceramics were higher than in the present study. However, the study design was different: in the present study, standardized and flat specimens obtained from natural teeth were used and the thickness of the enamel was controlled, whereas in the studies of Ioannidis and Maeder, the complete occlusal areas of molars were used, resulting in specimens with different geometries and luting areas. However, despite these methodological differences, higher breaking loads were found for aged 0.5 mm thick lithium disilicate ceramics which were luted to enamel (mean: 1692 N) compared to dentine (1165 N), too, which is in accordance with the results of the present study. For zirconia, the cited studies could not provide any information for specimens luted to enamel. They were not able to produce ultra-thin zirconia veneers with their materials. In our study the production of these discs was possible. In the present study, several 0.5 mm thick ceramic discs luted to dentine failed during artificial ageing: two in the zirconia group and 5 in the lithium disilicate ceramic group. In a study of Yazig et al. it was found, that artificial ageing of lithium disilicate ceramic resulted in cracks in 23% of the specimens. Belli R. et al. showed, that the strength degradation after cyclic loading was up to 53.4%, indicating a susceptibility to ageing. In the present study the focus is on the failure load. For this reason no fracture analysis was performed on the fractured specimens.
In general, zirconia performed better than lithium disilicate. However, both the mechanical properties and the effect of artificial aging on the materials are different. Having a closer look at these differences, three major aspects might be responsible:

1. One possible explanation for this finding might be the fact that the flexural strength of zirconia is superior compared to lithium disilicate.\textsuperscript{26} Even the “newer” materials 4Y-partially stabilized zirconia and 5Y-partially stabilized zirconia (e.g. Katana STML) show higher flexural and flexural fatigue strength compared to lithium disilicate ceramic.\textsuperscript{27}

2. The elastic mismatch between the ceramic and the cement results in tensile stress at the lower surface of the ceramic (ceramic – cement interface).\textsuperscript{28} This stress leads to failures in lithium disilicate ceramic specimens due to the reduced mechanical properties of the material (compared to zirconia).

3. Furthermore, artificial aging might have an effect on the cement, also,\textsuperscript{29} leading to a degradation in bond strength (to dentin) resulting in a redistribution of stress in the ceramic material. This could have a more pronounced negative influence on the lithium disilicate ceramic due to its reduces flexural strength.

Some limitations of the present study have to be kept in mind when interpreting the results: In the present study, exclusively specimens with enamel or dentine were used, although in clinical situations often a combination (a small band of enamel surrounds a large area of dentine in worn teeth) can be found. This approach was chosen in order to assess the effect of dentine and enamel separately without any interaction. Additionally, standardized specimens were used instead of real occlusal ceramic veneers. This approach was necessary to avoid the influence of any geometric aspects on the results. Furthermore, deeper parts of the dentine were used, presenting a lower elastic modulus,\textsuperscript{13} simulating a worst-case scenario. Finally, the time of water storage was short and,
consequently, the effect of hydrolysis on the fracture strength, which might be more relevant in zirconia specimens\textsuperscript{30,31} could not be assessed.

5. Conclusions

Both zirconia and lithium disilicate ceramics showed reduced breaking loads after artificial ageing but might be used for 0.5 mm occlusal veneers when luted to enamel. When 0.5 mm occlusal veneers should be luted to extremely worn teeth on dentine, zirconia might be more suitable. However, as the effect of hydrolysis has not been assessed in the present study in detail, the long term success of zirconia restorations should be examined further.

Conflict of interest: The authors declare that they have no conflicts of interest.

Acknowledgement

The study was supported by Kuraray Europe GmbH, Germany.

References


### Tables

#### Katana after aging

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<tr>
<th>Specimen</th>
<th>K1E0.5D2.5</th>
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<th>K0.5E1D2.5</th>
<th>K0.5D3.5</th>
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<tbody>
<tr>
<td>Median</td>
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<td>605</td>
<td>922</td>
<td>327</td>
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<tr>
<td>IQR</td>
<td>208</td>
<td>97</td>
<td>267</td>
<td>269</td>
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<tr>
<td>fractured</td>
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#### IPS e.max after aging

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<th>IPS0.5E1D2.5</th>
<th>IPS0.5D3.5</th>
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<td>Median</td>
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<tr>
<td>IQR</td>
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#### Katana un-aged

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<td>Median</td>
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<td>IQR</td>
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#### IPS e.max un-aged

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<th>IPS0.5E1D2.5</th>
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<tr>
<td>IQR</td>
<td>168</td>
<td>74</td>
<td>178</td>
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**Table 1** - Results of the specimen testing. The median and the interquartile range (IQR) were calculated. Abbreviations are described in Fig. 1 and 2.
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<table>
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Table 2 - Results of the Mann-Whitney U-Test of the un-aged and groups after ageing.

Abbreviations are described in Fig. 1 and 2.
Figures

**Fig. 1** – (a) The device for the initial cut of the dentin plates. The tooth is fixed with silicon (purple). The parallel slices in the tooth can be seen. (b) After this the plate is separated from the tooth and mounted to the next device. It is for the creation of the round tooth discs. (c) A cross section of the finished enamel-dentin (ED) tooth disc with bonded material (light blue).

**Fig. 2** – (a) Cross section of the cementation device: The tooth disc from Fig. 1 c is placed in the middle. After the combination of the blue and red element, the weight (yellow) was placed above. (b) Cross section of a tooth disc and the indenter (red). The plate holder is displayed in blue. (c) The fixation device for the specimens in the thermocycler. The specimens were placed between the red and blue parts. The parts were fixed by clamps (grey).
Fig. 3 - Whiskers-Boxplots diagram of the initial breaking loads (n=8 per group) in un-aged specimens.
Fig. 4 - Whiskers-Boxplots diagram of the initial breaking loads (n=8 per group) in aged specimens. Groups with a smaller specimen count are numbered above the boxplot bars.