

Influence of Heat Treatment and Veneering on the Storage Modulus and Surface of Zirconia Ceramic

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ABSTRACT

Objectives: Glass-ceramic veneered zirconia is used for the application as fixed partial dentures. The aim of this investigation was to evaluate whether the heat treatment during veneering, the application of glass-ceramic for veneering or long term storage has an influence on the storage modulus of zirconia.

Methods: Zirconia bars (Cercon, DeguDent, G; 0.5x2x20 mm) were fabricated and treated according to veneering conditions. Besides heating regimes between 680°C and 1000°C (liner bake and annealing), sandblasting (Al₂O₃) or steam cleaning were used. The bars were investigated after 90 days storage in water and acid. For investigating the influence of veneering, the bars were veneered in press- or layer technique. Dynamic mechanical analysis (DMA) in a three-point-bending design was performed to determine the storage modulus between 25°C and 200°C at a frequency of 1.66 Hz. All specimens were loaded on top and bottom (treatment on pressure or tensile stress side). Scanning electron microscopy (SEM) was used for evaluating the superficial changes of the zirconia surface due to treatment. Statistical analysis was performed using Mann Whitney U-test ($\alpha=0.05$).

Results: Sintered zirconia provided a storage modulus E' of 215 (203/219) GPa and tan δ of 0.04 at 110°C. A 10%-decrease of E' was found up to 180°C. The superficial appearance changed due to heating regime. Sandblasting reduced E' to 213 GPa, heating influenced E' between 205 GPa (liner bake 1) and 222 GPa (dentin bake 1). Steam cleaning, annealing and storage changed E' between 4 GPa and 22 GPa, depending on the side of loading. After veneering, strong E'-reduction was found down to 84 GPa and 125 GPa.

Conclusions: Veneering of zirconia with glass-ceramic in contrast to heat treating during veneering procedure had a strong influence on the modulus. The application of the glass-ceramic caused a stronger decrease of the storage modulus. (Eur J Dent 2011;5:191-198)

Key words: Zirconia, Veneering, Storage Modulus, CAD CAM.

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INTRODUCTION

Zirconia fixed partial dentures (FPDs) are used as alternatives for metal supported dental restorations. For protection and aesthetic aspects, the zirconia core is veneered with glass ceramics, which were adjusted in thermal expansion coefficient (TEC) and firing temperature (FT), e.g.. Insufficient tuning of these aspects may lead to fracture of the veneering under clinical conditions.¹⁻³

During baking of the veneering, small differences in TEC generally cause tensile stress on the ceramic, improving the bond between glass-ceramic and zirconia core. Veneering with in comparison to zirconia ($E \approx 200$ GPa) "weaker" glass ceramics ($E < 100$ GPa) may result in reduced stability of the FPD,⁴ but contradicting opinions were published.⁵ The location, whether the veneering is placed under pressure (on top) or tensile stress (bottom) is recorded to be of significant influence on the strength of the restoration.⁶ Stress in the veneering may cause manifold failures: cracks, which evolve in the glass ceramic may run in between the border between veneering and core (interfacial chipping), in a superficial layer of the veneering (chipping) or even may jump over into the core (fracture). Tensile or compressive stress may develop due to different visco-elastic relaxation mechanisms in two-layer systems.⁷ The application of opaquer as a stress-brake improved the bonding between zirconia and layering glass ceramic, but the combination of opaquer and press veneering reduced bonding results.⁴

As well as the application of veneering material may cause weakening of a restoration, heat or superficial treatments may influence zirconia ceramic. Temperature loadings up to 250°C ⁸ are regarded without any influence on the structure of zirconia, but the question arises, whether heat treatment during veneering may cause variations. According to the manufacturers' instruction the zirconia restoration is baked for applying liner, shoulder, dentin, glaze/stain, correction or final shoulder with decreasing temperature. During veneering process, the zirconia framework is subjected to a graduated thermal treatment between 1000°C and 680°C .

Partly, sandblasting of the surface is recommended before veneering or cementation of the FPDs. This procedure modifies the surface after

milling and reduces superficial defects or milling traces, but it is also assumed to be responsible for damaging the surface and causing microcracks. A superficially induced damage is presumed to cause tetragonal (t) to monoclinic (m) – transformation, which may run into the bulk material.⁹ Annealing at $1000^\circ\text{C}/1$ h is supposed for healing these damages and for improving strength and Weibull modulus of zirconia.¹⁰

These techniques may modify the zirconia surface, but it is not distinct whether the described treatments during veneering may modify zirconia strength and visco-elastic behaviour. The application of the veneering, which results in the formation of a bi-layer systems may have a strong influence on the elasticity of the whole restoration. The modulus of elasticity was shown to be an important factor for the strength evaluation of a multi-layer system.^{11,12} Dynamic mechanical analysis (DMA) is used for evaluating the changes in visco-elastic properties, subjecting the specimens to a defined, forced sinusoidal oscillation measuring reduced and deferred output. The differences in phase angle and force amplitude between input and output were used for calculating the storage modulus, which correlates with the modulus of elasticity. DMA is a complex analytical method, which allows for determining even small differences in modulus. The aim of this study was to investigate the influence of firing, sandblasting and storage on the storage modulus of zirconia. The influence of glass-ceramic press or layering veneering on the storage modulus should be investigated.

MATERIALS AND METHODS

Rectangular bars of the zirconia core material Cercon base (DeguDent, D) were milled with a water-cooled cutter (Leica SP1600, Bensheim, Germany) and sintered (Cercon Heat) to final dimensions (height: 0.5 mm, width: 2 mm, length: 20 mm). The height of 0.5 mm was used as representative for a standard thickness coping.

All specimens were divided into groups of three specimens each (Table 1). Blank zirconia cores (#C) were subjected to a standard veneering temperature program of a zirconia ceramic core without applying veneering ceramic. Temperatures and times are provided in the Table 1.

Groups C_{acid, water, anneal} were formed for investi-

gating the influence of heat treatment and storage on a core without veneering. Group C_{anneal} provides annealing of the sandblasted core. Group C_{water} investigates the samples after 90d storage in water and group C_{acid} in acidic acid (25%). In all groups no veneering material was applied.

In group LT zirconia specimens were investigated with additional veneering ceramic (Cercon Kiss, Degudent, G) using layering technique (=LT). Group PT was designed for investigating the influence of a ceramic veneering, which was applied in pressable technique (=PT) (Cercon Xpress, surface treatment 50 µm/0.2 MPa glass pearls; Degudent, G). Measurements in both groups PT and LT were performed using a core thickness of 0.5 mm (although the specimens were thicker with additional veneering) and the real thickness core with veneering (thickness 0.5 mm + veneering) as a direct comparison.

For investigating the superficial influence of each treatment, scanning electron micrographs (Field emission- SEM Phillips magnification: 30,000x) were made. The surface roughness was examined (Perthometer SP6; Perthen, G) and energy dispersive x-ray spectroscopy (EDX/SEM Phillips, 30 KV) for analysing the ceramic composition was performed.

All groups were investigated in three-point design, where the specimen is supported on two edges and the end of the push rod applies load centrally from the top (amplitude: 20 µm, dynamic load: 6 N, static load: 0.2 N, Frequency: 1.66 Hz). The distance between the two edges was 10 mm. Before testing, dimensions of the bars were determined with accuracy up to 0.01 mm (micrometer gauge). All samples were subjected to a temperature program between 25°C and 180°C in air atmosphere with a heating rate of 10 K/min (Dynamic mechanical testing device DMA 242, Netzsch, G). All measurements were repeated twice from both sides, investigating differences of surface treatment/veneering on top (pressure zone) or bottom (tensile zone). E' was determined at a clinical relevant mouth temperature of 37°C.

The complex modulus of elasticity ($E^* = E' + iE''$) in three-point-bending configuration is calculated as follows:

$$E^* = (l^3 \times F) / (4 \times w \times h^3 \times a^*), \{1\} \text{ where:}$$

$$E^* = \text{complex elasticity modulus [Pa]}$$

$$E' = \text{storage modulus [Pa]}$$

$$E'' = \text{loss modulus [Pa]}$$

$$a^* = \text{complex dynamic displacement [mm]}$$

$$F = \text{dynamic load [N]}$$

$$h = \text{sample height [mm]}$$

$$l = \text{bending length [mm]}$$

$$w = \text{sample width [mm]}$$

Tan δ is calculated as the relation between E' and E''. The application of veneering (group PT/LT) on zirconia core changed the mono-layer system to a bi-layer, where the influence of the thickness of both layers has to be regarded in the calculation of E'. According to formula {1}, the height of the specimen has to be considered with the power of three. Coherent, E' of the bi-layer system is further labelled E'_{bi}. For estimating the influence of the veneering glass-ceramic on the E'_{bi}, we investigated the bars in relation to the real core thickness (0.5 mm + height of veneering ceramic) as well as on the original core height (0.5 mm, E'_{bi} "effective").

Zirconia may show strong variations due to the Weibull strength distribution according to fabrication, treatment or surface conditions. Therefore, the influence of the treatment was investigated on every single bar by calculating E' or the difference ΔE' after relevant treatment in relation to the situation after sintering.

Median and 25-/75-percentiles were calculated and pairwise tests for statistics were carried out using Mann-Whitney-U-Test at a level of significance P ≤ 0.05.

RESULTS

The results and figures show the storage modulus (E') at a temperature of 37°C. After sintering median (25%/75%) E' was 215 (203/219) GPa. The storage modulus decreased with increasing temperature (from 30°C to 180°C) about 10%. For sintered zirconia a tan δ of 0.04 was found at about 110°C (Figure 1). For further treatments no shift of tan δ was found. The thickness of the tested specimens was between 0.49 ± 0.03 mm.

The superficial appearance of zirconia changed from the sintered state and milled state over sintering, sandblasting, liner, shoulder bake, dentin bake and glaze/stain (Figure 2). Sandblasting with Al₂O₃ caused a small reduction of median E' to 213 GPa. The application of liner bake 1 and 2 resulted in a storage modulus of 205 GPa and 209

GPa. Shoulder bake, as well as both dentin bakes and stain bake, increased E' , whereas maximum E' was found after dentin bake 1 (222 GPa). A subsequent reduction to $E'=209$ GPa could be determined with correction bake. With final bake, E' reached the level after sintering (216 GPa). Steam cleaning changed E' by about 4 GPa. None of the changes was significant ($P>.075$) (Figure 3).

Neither annealing (5 GPa) nor a 90d-storage in acid (2 GPa) or water (7 GPa) had a significant influence on $\Delta E'$ (Figure 4) when the specimens were investigated with the treated side on the top (pressure zone). Turning the specimens round and placing the treatment on the support side under tensile stress caused a $\Delta E'$ of -6 GPa (annealing), 8 GPa (90 days acid) and 22 GPa (90 days water).

The application of veneering resulted in a highly significant ($P>.75$) reduction of E'_{bi} . With veneer-

ing, the thickness ratio veneering:core of the bars was 1.2:1. The application of liner increased thickness of about 0.05 ± 0.008 mm. The influence of the glass-ceramic veneering on the bi-layer system was stringent: under optimal fabrication conditions, the storage modulus E'_{bi} decreased to values between 84 and 125 GPa for both types of veneering (Figure 5). No significant differences were found between layering and press ceramic application and when the veneering was placed on the bottom of the bar. Specimens were excluded from further evaluation, when defects due to fabrication (inclusions, air) or insufficient bonding between the two ceramic layers caused a significant reduction of E'_{bi} below E' of the veneering ceramic. Influenced by the fabrication process, E' decreased to values between 57 and 62 GPa for both types of veneering (Figure 6; below). In this case, when the veneering was placed on the bottom of the bar, further decrease of E'_{bi} was found. The application of veneering in press-technique caused a higher decrease of E'_{bi} in comparison to the layer application.

Effective E'_{bi} increased, when ignoring the increase of thickness due to veneering material and relating E'_{bi} on core thickness (0.5 mm). The plain increase in thickness due to application of liner, dentin or stain masses caused an increase of E'_{bi} up to 268 GPa (liner), 592 GPa (dentin) and 670 GPa (stain) with the veneering on the top side of the

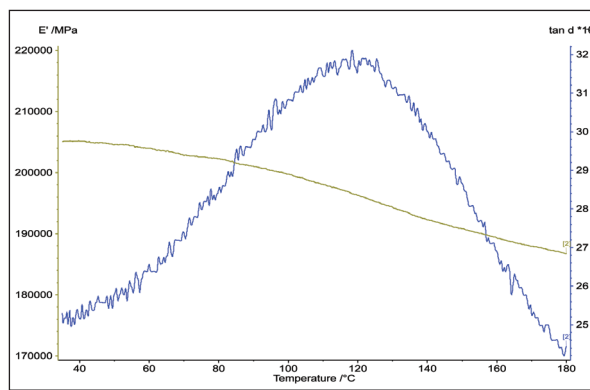


Figure 1. Storage module E' [GPa] and $\tan \delta$ of sintered zirconia after sintering (example).

Table 1. Study overview and classification of the different treatments (X indicates the performed treatments).

Groups		C	LT	PT	CAnneal	Cacid	Cwater
		Layering technique		Press technique	Annealing	90 d acid	90 d water
Material		Core	Core + veneering			Core	
Bar thickness (mm)		0.5	0.5 and 0.5 + veneering			0.5	
After	Temp [°C] / Time [min]						
Sintering		X	X	X	X	X	X
Al ₂ O ₃ (110 μm/0.25 MPa)		X	X	X	X	X	X
Steam cleaned		X	X	X			
Liner 1	970/1	X	X				
Liner 2	960/1	X					
Shoulder bake	850/1	X					
Dentin first bake	830/1.5	X	X	X			
Dentin second bake	820/1.5	X					
Glaze/Stain	800/1	X	X	X			
Correction	680/1	X					
Final shoulder	680/1	X					
Annealing	1000/				X		
Water storage 90 days							X
Acid storage 90 days						X	

bar. Smaller increase up to 248 GPa (liner), 391 GPa (dentin) and 422 GPa (stain) was found, when the veneering was applied on the bottom. Main differences were caused by the application of dentin masses, whereas stain ceramic had no further effect (Figure 6, above).

Surface roughness varied between 0.1 and 0.2 μm , but no significant differences between the various heat treatments were found. EDX analysis revealed differences only after air abrading. Al-particles (1.6 wt%) could be detected on the zirconia surface. All other treatment had no EDX-visible effects.

DISCUSSION

The storage modulus E' of 216 GPa, which could be determined with DMA correlates with the modulus of elasticity of 210 GPa in literature.¹³ E'

showed a decrease of about 5% in dental application temperature range between 5°C and 80°C. This decrease is associated with a dimension change of 20 μm and with a damping response, which is shown by a dipole peak ($\tan \delta$ of 0.04) at about 120°C. This phenomena is described as a stress induced reorientation of elastic dipole moments ($\text{Yzr}'\text{Vo}''$)^{14,15} and may be a helpful tool to differentiate between different types of dental zirconia.

Although SEM figures (Figure 2) showed changes of the zirconia surface with different treatments, only small variations of E' could be determined. Sandblasting, which showed the highest optical superficial changes and additional Alumina on the surface, as well as steam cleaning had no effect on storage modulus. Roughness due to surface treatment did not change significantly.

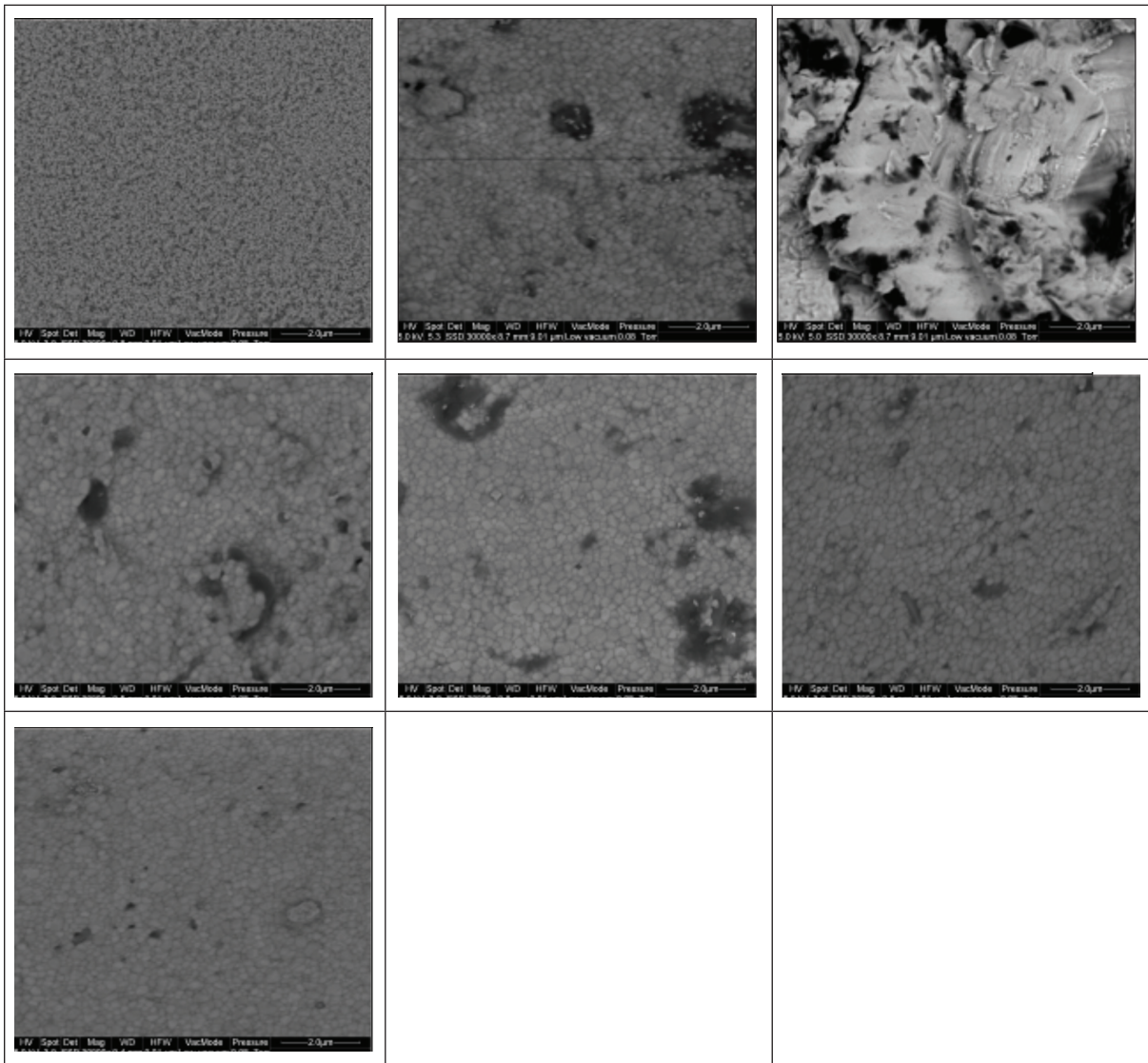


Figure 2. SEM figures of the zirconia surface: first row: milled, sintered, sandblasted, second row: liner bake, shoulder bake, dentin bake, third row: glaze/stain bake.

The simulated liner bake (970°C) reduced median E' about 5%. This results correspond with a reported 5%- decrease of flexural strength with heat treatment,¹⁶ which is caused by t-m transformation. Subsequent temperature programs with temperatures down to 830°C (dentin bake) increased median E' again, whereas further heat treatments down to 680°C had only small influence on E' . Sundh et al¹⁷ showed, that the temperature of heating treatment of zirconia hat an influence on the fracture strength of fixed partial dentures. With treatment above 900°C fracture strength halved, whereas treatment of about 750°C caused a reduction of only about 23%. How-

ever, same authors¹⁷ found no different fracture results, whether a zirconia core was veneered or not.

It has been described, that sandblasting improved the mean strength of zirconia in expense of its reliability,¹⁸ but we found only small, non-significant changes of E' . It was supposed that particle abrasion may cause a superficial t-m transformation,¹⁶ creating a layer of compressive strength, which works against the before induced flaws.^{9,18} Flaws, which may not reach deeper than the compressive zone, may explain the strength increase with abrading. Longer flaws -in contrast- would result in weakening of the material. However flaws on the tensile loaded surface may grow to slow crack growth mechanisms.^{13,19} Abrading caused high deviation of the flexural strength and reliability, what might affect clinical use.^{9,16,18}

No influence of E' after storage in water or acid could be determined, when the sandblasted surface was tested in pressure zone. Turning round the bar and subjecting the sandblasted and stored surface to tensile loading resulted in partly different results. Besides storage in acid showed only small changes, storage in water resulted in a median change of about 10% of E' . It was described that besides increased temperature (about 250°C) and high grain size, water/humility is caused responsible for transformation processes in zirconia ceramic. Water forms superficial zirconium hydroxides due to water chemisorption and causes strain energy accumulation and m-t transformation. On the other side water might react with yt-

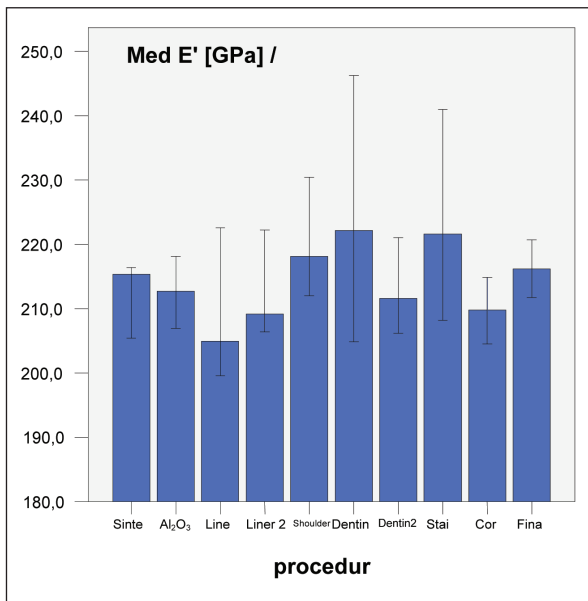


Figure 3. Change of E' [GPa] in relation to E' after sintering; influence of the surface/heat treatment (group A).

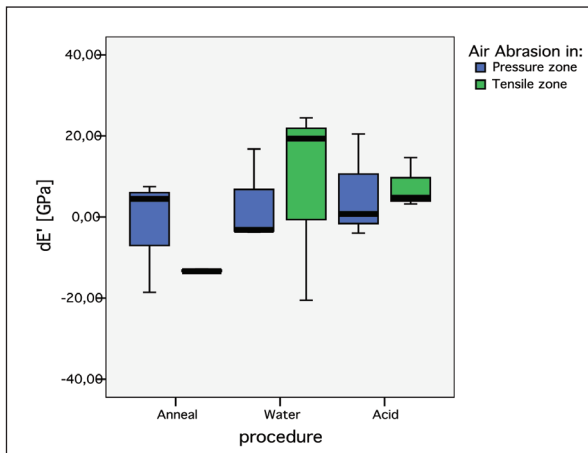


Figure 4. Change of storage modulus (dE' [GPa]) in relation to E' after sintering; influence of aging conditions (annealing, 90 days storage in water / acid); (specimens were loaded treatment in pressure or in tensile zone).

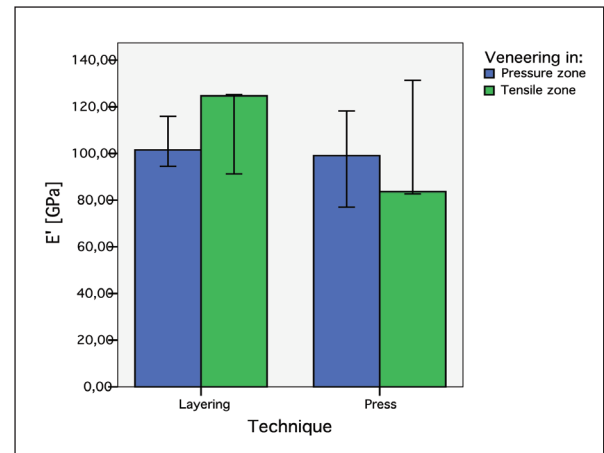


Figure 5. E' [GPa] after veneering in layer- or press technique (specimens were loaded treatment in pressure or in tensile zone).

trium forming yttrium hydroxide, which depletes the stabilization causing m-t transformation.⁹ Ardlin et al²⁰ described that storage in ringer solution had no effect on zirconia. In contrast to wet storage, storage at 120°C for 120 hrs caused only small variations⁸ and even after 30 months no substantial change in bending strength could be found.²¹ Especially aging under low temperature had no effect on the flexural strength of zirconia bars.⁹ Aging is supposed to influence the amount of yttrium, which is responsible for the grain size, which in turn influences zirconia transformation.²²

The nearly doubled thickness of the testing bar with veneering led to a seeming strong increase of E'bi with extreme variations up to 100%, whether the veneering placed on top or bottom of the bar. The real thickness of the specimens, E'bi was reduced about 60% with veneering, reaching the modulus of the individual veneering ceramics. Under optimal manufacturing conditions, small differences were found between layering and press

ceramics, which may be put down partly to the application of liner for the layering technique or the recommended glass-pearl treatment before performing the press method. Dimensional influences especially due to varying edge stability may not be excluded. Specimens with defects or insufficient bonding between the two ceramic components caused an extreme decrease of E'. The results indicate, that the veneering with a "weak" ceramic had a strong influence on the whole specimen. This is in accordance to microtensile investigations,⁴ finite element analysis²³ or bending tests,⁶ where the veneering had predominant effects on the properties. These results are significant for the fabrication of bridges, where one source for defect is the fracture of the pontic on the tensile side of the connector.^{24,25} Chipping may be avoided by considering modulus and veneering thickness, because both parameters have influence on the stress ability of the restorations.¹² Whereas the increase of core thickness (maintaining the total thickness core + veneering constant) is supposed not to improve the stress of the bi-layered system,²⁶ the change in the modulus of elasticity in a bi-layer (or even multi-layer system taking cement or tooth substance into account) may influence the strength of restorations.^{27,28} Differences in modulus cause variation of energy absorption/dissipation and may in the end cause chipping, interfacial chipping or fracture.

CONCLUSIONS

The veneering of zirconia with glass-ceramic materials may have a strong influence on the modulus of the dental restorations. Long term storage in water may contribute to further deterioration. Especially for the application of the glass-ceramic in tensile stress weakening of fixed partial dentures should be expected. Heat treatment of the zirconia core during firing of the veneering had no significant influence on the storage modulus.

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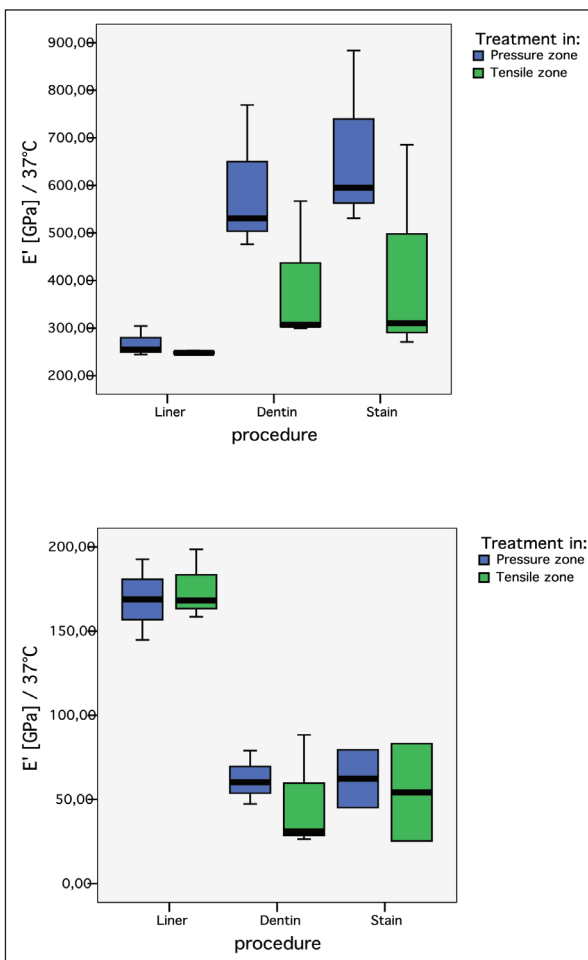


Figure 6. Storage modulus (E' [GPa]): influence of applied liner, dentin or stain masses; above: related to core thickness- below: related to thickness core + veneering (specimens were loaded treatment in pressure or in tensile zone).

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