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ORIGINAL PAPER

The Effect of Heat Treatment and Feldspathic Glazing on Some Mechanical Properties of Zirconia

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Abstract
Purpose In dental practice, zirconia substructures for crowns and fixed partial dentures are veneered with feldspathic porcelain for better aesthetical properties. The purpose of this study was to evaluate the effect of heat treatment during the veneering process on the mechanical properties of zirconia.
Methods 105 zirconia (ICE Zirkon, Zirkonzahn, Italy) disc shaped specimens were divided into seven groups (n=15/group) and sintered at 1500 °C. Control specimens (group 1) were left as such. Specimens in the study groups were: 2) one heat treatment in a porcelain firing furnace; 3) a heat treatment with two firing cycles; 4) one heat treatment with a thin coating of feldspathic glazing on the tension side; 5) two heat treatments with a thin coating of feldspathic wash and glazing on the tension side; 6) one heat treatment with a thin coating of feldspathic glazing on the compression side; 7) two heat treatments with a thin coating of feldspathic wash and glazing on the compression side. Biaxial flexural strength and surface microhardness of the specimens (diameter 19 mm, thickness 1.2 mm) were measured. The effect of heat treatment and feldspathic glazing on the phase transformations of zirconia was determined by XRD-analysis. The data were calculated using ANOVA-analysis.
Results Repeated heat treatment did not influence the mechanical properties of zirconia (p>0.05). No difference (p>0.05) was found in terms of the surface microhardness between the groups. Some transformation from the tetragonal to the monoclinic phase was seen on the surface of the specimens after heat treatment and feldspathic glazing coating.
Conclusions Repeated heat treatment does not affect the strength of zirconia milled in the green-stage form.

Keywords Zirconia · Heat treatment · Flexural strength · Feldspathic glazing porcelain

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1 Introduction

Yttrium partially stabilized zirconium dioxide (ZrO_2), zirconia, used for the substructures of dental crowns and fixed partial dentures is veneered with feldspathic porcelain for natural tooth appearance. The substructure and veneering porcelain form a bi-layered structure, where the material that is placed on the bottom surface (tension side) has an impact on the strength due to crack initiation and propagation [1, 2]. It also dictates the reliability and fracture mode of the specimen [3]. In a study of a bi-layered porcelain/zirconia material combination it was found that by increasing the thickness of zirconia, the flexural strength of the tested specimen increased [2].

Zirconia is a polymorphic material and occurs in three forms: monoclinic, tetragonal and cubic. The monoclinic phase is stable from room temperature to 1170 °C, tetragonal in the range 1170–2370 °C and cubic over 2370 °C. The phase transformation in pure zirconia from the tetragonal to the monoclinic phase occurs during cooling and is associated with a volume expansion of 3–5%. By the addition of some stabilizing oxides, such as

Y_2O_3, zirconia can also be stabilized in the tetragonal phase at room temperature [4]. Transformation in zirconia from the tetragonal to the monoclinic phase can be initiated by stress, temperature and surface treatment [5–7]. Even if some phase transition occurs, it has a minor effect on the mechanical properties of zirconia [8–10].

Zirconia undergoes a heat treatment cycle as part of the veneering porcelain firing when manufacturing dental crowns and fixed partial dentures. The feldspathic veneering porcelain is fired at a temperature in the range 700–900 °C. Chevalier et al. [11] found that heat treatment of the zirconia at 1200 °C releases residual stresses without affecting the surface relief. In recent studies it has been found that the heat treatment by the veneering process lowers the strength of zirconia, which has been milled and ground in the sintered form [12, 13]. When grinding sintered zirconia, the milling and grinding caused a phase change from tetragonal to monoclinic on the surface of the zirconia. The results indicated that heat treatment influences the surface of the ceramic material by reducing the amount of monoclinic zirconia grains, which is suggested to result in a lower strength. Vigolo and Fonzi [14] found that the veneering porcelain firing cycle or the veneer glazing cycle did not affect the marginal fit of the fixed partial dentures. Whereas Dittmer et al. found in a recent study that stresses and distortions that occur due to the veneering process may influence the marginal and internal fit of zirconia dental crowns and fixed partial dentures [15]. In another study of glazing dental feldspathic porcelain it was found that glazed porcelain discs had lower flexural strength than porcelain discs that were just polished. The strength of discs fired once was significantly greater than the strength of twice-fired discs [16].

The effect of heat treatment of zirconia that has been milled in the green-stage form has not yet been evaluated. The purpose of this study was to determine the effect of heat treatment by the veneering porcelain firing process and the effect of coating the zirconia substructure material with a thin layer of feldspathic glazing porcelain on the mechanical properties of yttrium partially stabilized green-stage zirconia. The hypothesis in the study was that the heat treatment of the veneering process affects the material combinations strength.

2 Materials and Methods

2.1 Preparation of the Samples

Yttrium partially stabilized green—stage zirconium dioxide, zirconia blocks (ICE Zirkon, Zirkonzahn, Sand in Taufers, Italy) were cut into disc shaped specimens with a low speed diamond saw (Leitz 1600). After cutting a total of 105, the specimens were ground with 800 and 4000 grit (FEPA) silicon carbide grinding paper (Struers, Copenhagen, Denmark) to a thickness of 1.6 mm. The specimens were then divided into seven groups (n=15/group). The ceramic disc shaped specimens were sintered at 1500 °C in a sintering furnace (Zirkonzahn) using a temperature rise time of 3 h and keeping the temperature at 1500 °C for 2 h before cooling down. Control specimens (group 1) were left as such. Specimens in the study groups were subjected to the following: 2) one heat treatment in a porcelain firing furnace according to the porcelain firing process; 3) two repeated heat treatment cycles in a firing furnace according to the firing process; 4) one heat treatment with a thin coating of feldspathic glazing porcelain (ICE Zirkon keramik) fired on the specimens, tested glazing on the tension side; 5) two heat treatments with an initial thin coating of feldspathic wash porcelain and a second thin coating of glazing fired on the specimens, tested wash and glazing on the tension side; 6) one heat treatment with a thin coating of feldspathic glazing porcelain fired on the specimens, tested glazing on the compression side; 7) two heat treatments with an initial thin coating of feldspathic wash porcelain and a second thin coating of glazing fired on the specimens, tested wash and glazing on the compression side (Table 1). Heat treatments were made in a porcelain furnace (Vacumat 200 Vita Zahnfabrik, Bad Säckingen, Germany). The heat treatment program started at 400 °C with 6 min preheating. Then the temperature was raised at 55 °C per minute with an applied vacuum. Specimens were kept at the final temperature of 820 °C for one minute before cooling started. Specimens in the second group were heat treated at 820 °C without coating. Specimens in the third group were heat treated twice at 820 °C without coating. Specimens in the fourth and sixth group were heat treated at 820 °C with a thin coating layer of the mixture of the glazing (ICE Zirkon Glaze, LOT: MA50001A, ZirkonZahn) and stain liquid (Ice Stain Liquid, LOT: MFAA13_01, ZirkonZahn) over the discs. Specimens in the fifth and seventh group were heat treated twice at 820 °C. The first heat treatment of groups 5 and 7 were done with a thin coating layer of the mixture of dentin porcelain (ICE Zirkon Keramik Dentin D4, LOT: KA50053A, ZirkonZahn) and modeling liquid (ICE Build Up Liquid, LOT: MFAA14_01, ZirkonZahn) over the specimens. The second heat treatment was done with a thin coating layer of the mixture of glazing and stain liquid over the dentin porcelain. After firing the glazing the specimens were left as such and no grinding or polishing on the glazing surface was performed.

2.2 Mechanical Testing

The thickness and diameter of the sintered specimens was measured with a digital micrometer (Mitutoyo Ltd, And-

Table 1 Zirconia specimens were divided into seven groups according to their treatment

Group number and definition	n	First heat treatment and coating	Second heat treatment and coating	Note on biaxial flexural strength testing
1) Control	15	–	–	–
2) Plain specimens with one heat treatment	15	820 °C without feldspathic glazing	–	–
3) Plain specimens with two heat treatments	15	820 °C without feldspathic glazing	820 °C without feldspathic glazing	–
4) Specimens with glaze coating with one heat treatment	15	820 °C with thin coating of feldspathic glazing	–	Coating on tension side
5) Specimens with wash and glaze coating with two heat treatments	15	820 °C with thin coating of feldspathic wash porcelain	820 °C with thin coating of feldspathic glazing	Coating on tension side
6) Specimens with glaze coating with one heat treatment	15	820 °C with thin coating of feldspathic glazing	–	Coating on compression side
7) Specimens with wash and glaze coating with two heat treatments	15	820 °C with thin coating of feldspathic wash porcelain	820 °C with thin coating of feldspathic glazing	Coating on compression side

over, UK) before the fracture test. The mean thickness of the specimens was 1.2 mm (SD 0.1 mm) and the diameter 19 mm. According to the recommendations of ISO Standard 6872 [17], the bi-axial flexural strength test was used for determining bi-axial fracture strength values. Veneering the zirconia substructure material with a layer of porcelain would have led to a bi-layered material and likely to have hidden the impact of the actual heat treatment on the zirconia´s properties. Therefore, in order to get information on the possible change in zirconia material properties by heat treatment, instead of firing veneering porcelain a thin, less than 10 µm wash and glazing porcelain coating was used in study groups 4–7. The groups were tested using the ISO Standard and the method for actual bi-layered disc shaped specimens recommended by Hsueh et al. [18] was not justified. The specimens were tested dry at room temperature with a universal testing machine (Model LRX, Lloyd Instruments Ltd), where they rested on three symmetrically based balls and the load was applied to the centre of the top surface by the piston (diameter 1.60 mm) until fracture occurred (Fig. 1). The cross-head speed of the piston was 1 mm/min. The results were recorded with the PC-software (Nexygen, Lloyd Instruments Ltd, Fareham, England). The surface microhardness of the zirconia side of the specimens was determined by the indentation technique [19] to determine the Vickers hardness number (VHN) of the specimens using a load of 9.81 N. There were 20 randomly selected indentations per subgroup. The indentations were performed after the flexural strength test to avoid introducing defects in the specimens that could influence the flexural strength results.

2.3 Statistical Analysis

The data were calculated using one-way analysis of variance (ANOVA) followed by Tukey's HSD test at a significance level of $p<0.05$.

2.4 Scanning Electron Microscopy

Five specimens from each group with a coating on the surface (4–7) were ultrasonically cleaned and covered with carbon (Bal-Tec SCD 050, Sputter coater) before the imaging of the fracture surfaces with a scanning electron microscope (SEM) (Princeton Gamma-tech X-ray microanalysis). Magnifications of ×60, ×120, ×500 and ×5000 were used. Visual inspection of the fractured surfaces was carried out. The thickness of the coating layer of wash and glazing was measured from SEM—images. The thickness of the coating layer of glazing in groups 4 and 6 was 4.3 µm and the thickness of the

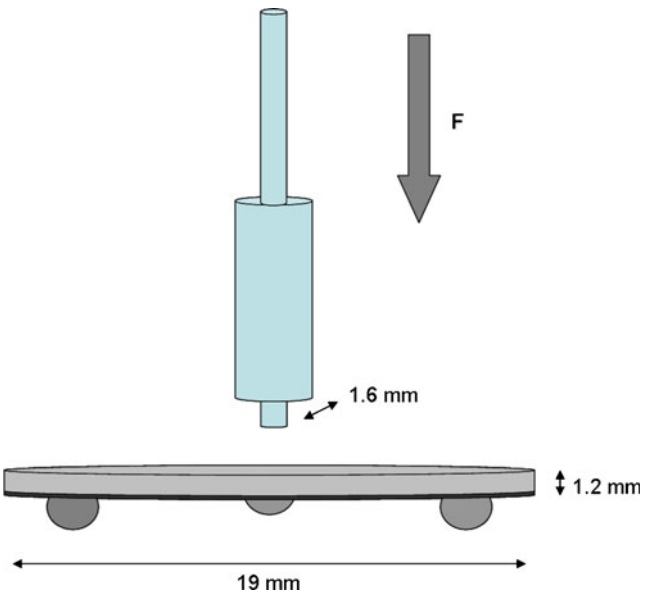

Fig. 1 Illustration of the bi-axial flexural strength testing machine. The tested specimens rested on three symmetrically based balls, and the load (F) was applied to the center of the top surface by the piston (diameter 1.60 mm) until fracture occurred. The dark line on the bottom of the specimen illustrates a thin coating layer of glazing on the tension side

coating layer of wash and glazing in groups 5 and 7 was 7.6 μm (Fig. 2a–b).

2.5 X-ray Diffraction Analysis

The effect of heat treatment on the phase transformations in zirconia discs was determined by X-ray diffraction (XRD) analysis (Philips PW 1830 Generator) using 40 kV and 30 mA and Cu Kα -1 radiation. XRD-analysis was performed on sintered and heat treated specimens (with and without a thin coating of wash and glazing on the opposite side of the specimen) and to sintered and twice heat treated specimens that were not yet mechanically tested. A sintered zirconia disc shaped specimen was used as a control.

The relative amount of monoclinic zirconia was determined according to the polymorph method recommended by Garvie and Nicholson [20] from the integral intensities of two monoclinic peaks M(III) and M(II$\bar{\text{I}}$) and one tetragonal peak T(III). The equation for the monoclinic phase is:

$$X_m = \frac{\mathrm{I_m(III)} + \mathrm{I_m}\left(\widehat{\mathrm{II}}\right)}{\mathrm{I_m(III)} + \mathrm{I_m}\left(\widehat{\mathrm{II}}\right) + \mathrm{I}_\tau(\mathrm{III})}.$$

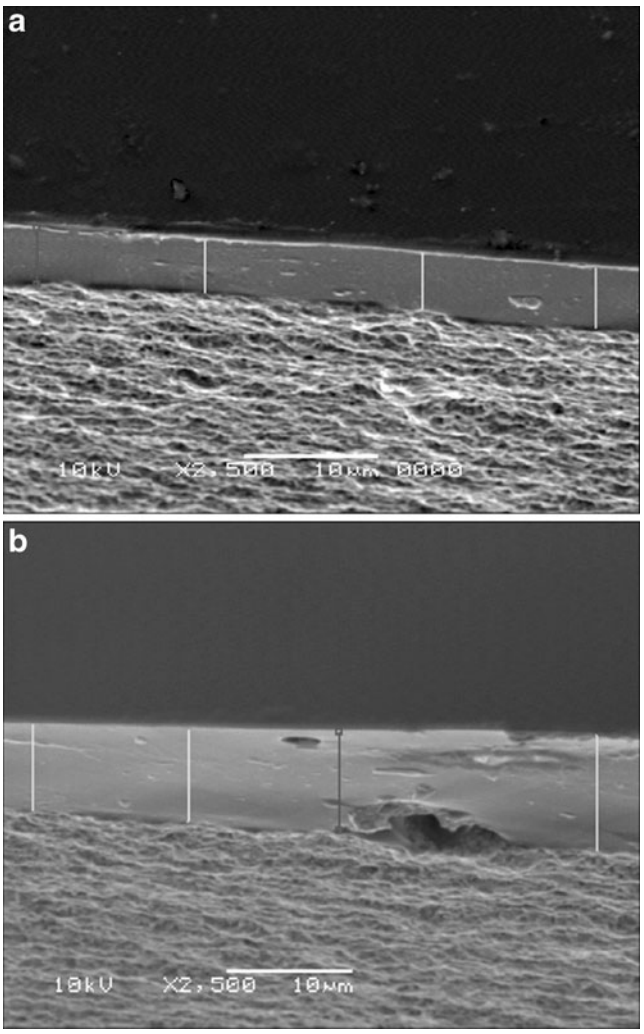

Fig. 2 Scanning electron microscope images with a magnification of 2500: Thickness of glaze coating layer (**a**), Thickness of wash and glaze coating layer (**b**)

3 Results

3.1 Mechanical Testing

A summary of bi-axial flexural strength, Vickers microhardness, maximum load and thickness of the specimens are given in Table 2. Flexural strength varied between 553.0 MPa and 1019.4 MPa (Fig. 3). One-way ANOVA- analysis revealed statistically significant differences in strength between the groups ($p<0.05$). Repetitive heat treatment slightly increased the biaxial strength, but the difference was not statistically significant ($p>0.05$). A thin coating of wash and glazing did not decrease the strength of the specimens when the coating layer was positioned on the compression side ($p>0.05$). When the specimens were tested with a coating of glazing or wash and glazing positioned on the tension side, the bi-axial flexural strength decreased ($p<0.05$). Loading curves by Nexygen software did not demostrate (Typo) any signs of existing pre-cracks in the material. No difference was found in terms of surface microhardness (VHN) ($p>0.05$) measured from the zirconia side of the specimens.

3.2 Scanning Electron Microscopy (SEM)

In the Scanning Electron Microscopy images some of the specimens showed minor de-lamination of the coating layer and the de-lamination were seen regardless of whether the coating was on the tension side or on the compression side of the specimen (Fig. 4).

3.3 X-ray Diffraction Analysis

The relative amounts of monoclinic zirconia for zirconia in the heat treated and coated groups were 1.2% (one heat treatment with glazing coating) and 1.7% (two heat treatments with wash and glazing coating). No monoclinic zirconia was found on the surface of the sintered control specimens or discs heat treated without coating (Fig. 5).

4 Discussion

This study was undertaken in order to demonstrate possible changes in the bi-axial flexural strength of bulk zirconia substructure material for dental crowns and fixed partial

Table 2 Bi-axial flexural, surface microhardness (VHN), maxiumum load and the height of the studied specimens. Different letters show the statistical difference in results between the groups

Parameters	Flexural Strength MPa (SD)	Stat diff.	Surface Microhardness VHN (SD)	Maximum load N (SD)	Stat diff	Height of the disc mm (SD)
Control	942 (142)	a	1420 (62)	846 (122)	bc	1.2 (0.01)
1 heat treatment	923 (170)	a	1443 (112)	872 (152)	bc	1.22 (0.02)
2 heat treatments	977 (98)	a	1382 (58)	798 (82)	cd	1.14 (0.03)
1 heat treatment = glazing on tension side	658 (98)	b	1399 (105)	705 (96)	de	1.27 (0.04)
2 heat treatments = wash + glazing on tension side	533 (80)	b	1437 (63)	625 (50)	e	1.32 (0.1)
1 heat treatment = glazing on compression side	1019.4 (105.3)	a	1526 (66.6)	1019.4 (110)	a	1.26 (0.02)
2 heat treatments = wash + glazing on compression side	918.5 (156.2)	a	1440 (117)	968.5 (171)	ab	1.29 (0.03)

dentures. The experimental heat treatment process of zirconia simulates the situation in a dental laboratory where feldspathic veneering porcelain is fired on zirconia substructures thus, causing heat treatment cycles for zirconia. Veneering is needed because zirconia as such does not meet the high aesthetic-cosmetic values for dental crowns and fixed partial dentures, and needs to be veneered to achieve higher results in terms of natural tooth appearance.

When using the piston-on-three-ball technique to measure biaxial flexural strength some contact stress is induced between the loading piston and the specimen. In the ring-on-ring technique such compressive stress does not affect the centre surface of the specimen. On the other hand, the compressive stress has not been shown to considerably affect the tensile stress of the specimen [18]. Guazzato et al. [1] noticed that as the load was increased, the crushing of the porcelain on the top surface occurred before the fracture of the core material. In our study, the piston-on-three-ball technique was used. The glazing coating layer was thin and pre-cracks were not observed during the loading process.

Using the thin coating on the specimens only a minor change of the dimensions of specimens were found compared to the situation where thicker porcelain layer would have been used in veneering. In the mechanical testing the samples with the coating on the tension revealed lower biaxial flexural strength. Before sintering bulk zirconia substructure materials were ground with 800 and 4000 grit grinding paper, which may have left some flaws

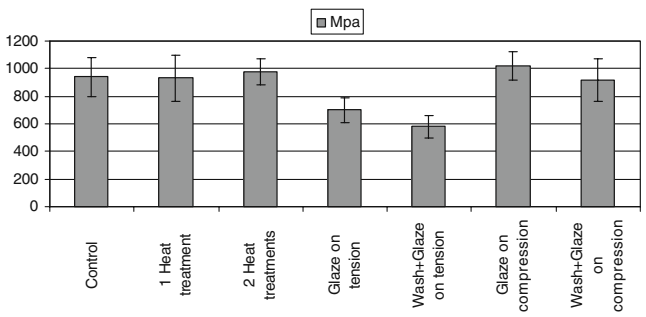

Fig. 3 Biaxial mean flexural strength (MPa) of the study groups; control, 1 heat treatment, 2 heat treatments, thin coating of glaze on tension side, thin coating of wash + glaze on tension side, thin coating of glaze on compression side, thin coating of wash + glaze on compression side

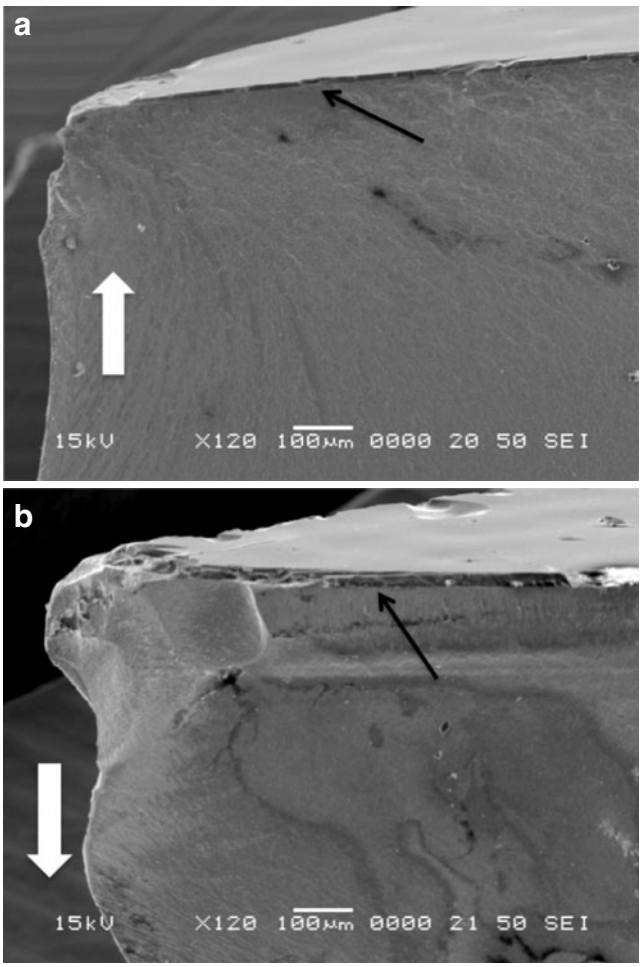

Fig. 4 Scanning Electron Microscope images of zirconia after heat treatment with thin coating of glazing and after biaxial flexural strength testing: **a** disc shaped specimen with coating on the tension side, **b** disc shaped specimen with coating on the compression side. The white arrow illustrates the direction of the loading and the black arrow illustrates the coating layer

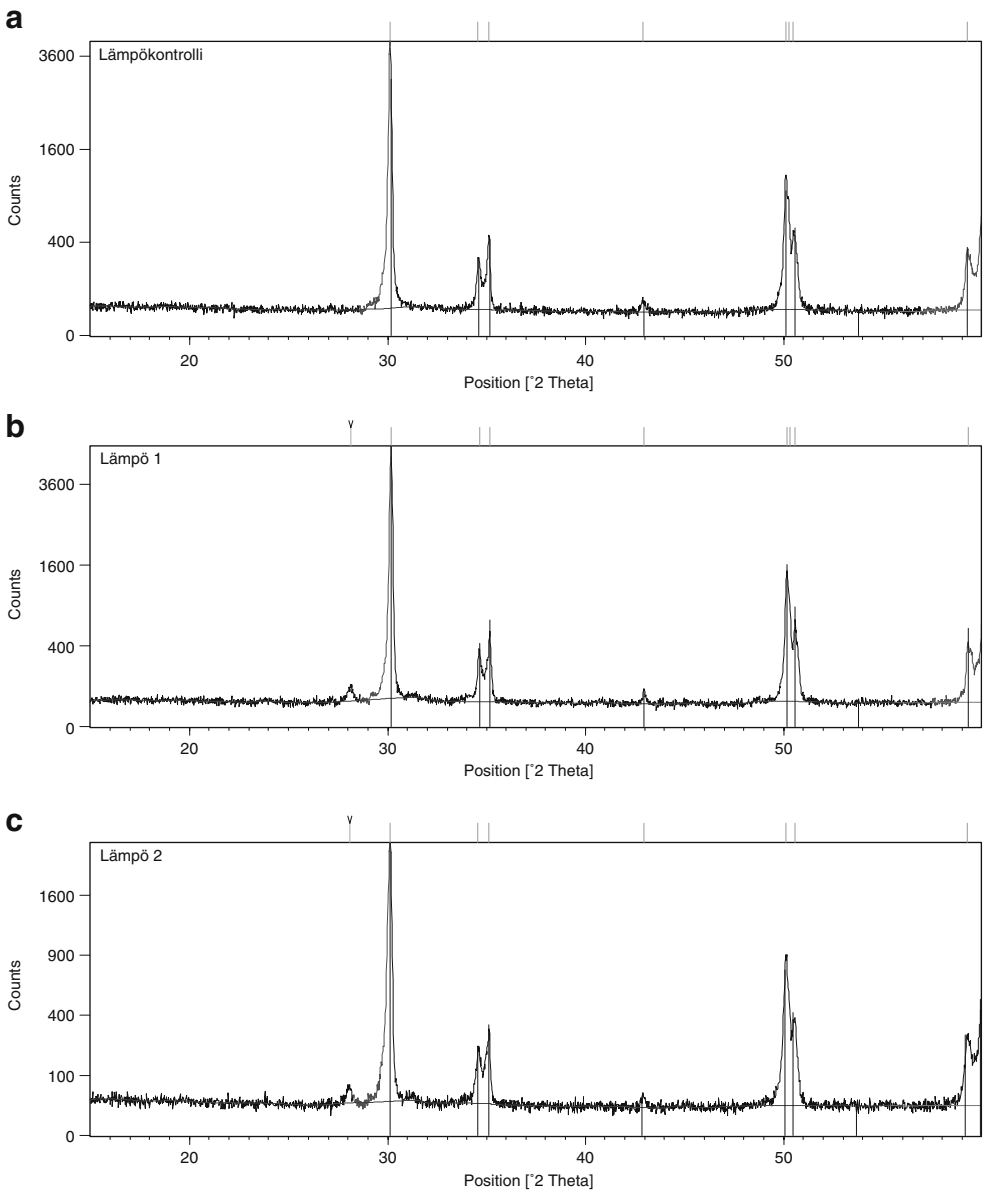

Fig. 5 Relative amounts of monoclinic zirconia determined by X-ray diffraction analysis: **a** normal control (X_M 0%), **b** one heat treatment (X_M 1.2%), **c** two heat treatments (X_M 1.7%)

to the surface of the specimens. During firing the glazing or wash layer on zirconia, melt wash or glaze behaved as a visco-elastic liquid and likely penetrated into flaws and microcracks. By cooling the viscosity increases and when bonded to another material with different thermal dimensional behavior the glass matrix of the wash or glaze liquid started to retain stresses when it changed into a solid [21]. The stress in the glazing and wash porcelain which filled the microcracks may have predisposed crack initiation and result in increased probability of fracture during loading of the restoration [22, 23].

After mechanical testing some de-lamination was seen on the wash and glazing coating although the coating layer was relatively thin. This supports the findings that the bonding between the zirconia substructure and the veneering porcelain might be insufficient and that the crack has a tendency to run along the porcelain/zirconia interface, causing de-lamination especially on disc shaped specimens [3, 24]. When veneered zirconia crowns have been tested with mouth-motion sliding-contact fatigue the cohesive fracture occurred in veneering porcelain and resulted in porcelain chipping [25]. This porcelain chipping from the substructure has also been seen as a failure in recent clinical studies [26, 27].

The influence of heat treatment on the properties of zirconia has also been reported in other studies. After air-particle abrasion, grinding or polishing the heat treatment of the zirconia specimen decreased the flexural strength of the material [28, 29]. This was explained by the compressive stresses caused by surface treatment such as sandblasting were released in the heat treatment and the mean flexural strength was influenced by sandblasting-induced defects in the material.

Øilo et al. [12] found that heat treatment of the veneering process lowered the flexural strength of zirconia, but multiple firing cycles did not further lower the strength. These materials were machined to restorations after final sintering. In our study, the zirconia material was ground in an un-sintered green-stage form. The effects of the firing procedure may depend on the form of manufacturing and our results support manufacturing substructures of fixed partial dentures and crowns from un-sintered green-stage material.

XRD-analysis was performed to see the possible phase change on the surface of the specimens. Small monoclinic peaks were seen on the surface of the heat treated specimens with wash and glaze porcelain. Earlier studies show that coloring of green-stage zirconia material does not cause any phase change but thermal cycling in 5–55 °C distilled water causes some changes [10, 30]. None of these phase changes had an effect on the mechanical properties of the zirconia material; however, the long term effects were not evaluated in this study.

According to the manufacturer of the material used in this study, the coefficient of thermal expansion (CTE) for ICE Zirconia was 10×10^{-6} K^{-1} and ICE wash and glazing 9.6×10^{-6} K^{-1}. When porcelain is fused to the bulk substructure material attention must be paid to the matching of the CTE values. A large mismatch between the porcelain veneer and core materials can cause residual stresses and result in chipping of the veneer [31, 32]. In our study, no large chippings but delamination of the wash or glaze coating were seen after mechanical testing in Scanning Electron Microscope.

In this study, there was no difference between the groups in surface microhardness. Phase changes from the tetragonal to the monoclinic phase on the surface of the specimens were minor (1.2–1.7%). Theoretically, a monoclinic phase transition on the surface could cause compressive stress to the outer layer of zirconia, which could increase surface microhardness. Future studies are needed to determine the surface properties of zirconia with more precise methods such as nanoindentation tests.

The hypothesis that heat treatment without additional glazing or wash porcelain weakens the zirconia bulk substructure material that has been ground in a green-stage form was rejected. The results showed decrease in strength when zirconia material was tested with additional thin coating of wash and glazing on the tension side, as seen in previous studies with bi-layered zirconia/porcelain specimens [1, 2]. In this study one or multiple heat treatments as such did not affect the material strength.

In conclusion, based on our results, repeated heat treatment does not affect the strength of the zirconia bulk substructure material which was milled in green-stage form but causes some transformation from tetragonal to monoclinic phase of zirconia when the coating of feltspathic wash or glaze porcelain is fired on zirconia specimens.

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