XRD² micro-diffraction analysis of the interface between Y-TZP and veneering porcelain: Role of application methods

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ABSTRACT

Objectives. The metastability of the tetragonal crystal structure of yttria partial stabilized zirconia polycrystalline (Y-TZP) ceramics is a basis of concern for dental restorations. Reactions between the porcelain and the Y-TZP framework may result in a reduction of the stability of the zirconia and interface bonding caused by a transformation from tetragonal to monoclinic crystalline structure during veneering.

Methods. XRD² micro-diffraction measurements were carried out on tapered veneered cross-sections of the interface area to generate locally resolved information of the phase content in this region. To get a high intensity X-ray beam for short measurement times a focussing polycapillary with a spot size of app. 50 μm was used to evaluate the porcelain zirconia interface.

Results. Under almost all conditions the phase composition of zirconia grains at the interface revealed both the monoclinic and tetragonal structure. These observations indicate that destabilization of the tetragonal phase of zirconia occurs at the interface during veneering with porcelain.

Significance. These results and their relevance to the long-term stability of the interface adhesion between zirconia and veneering porcelain as well as the tetragonal to monoclinic crystal transformations at the interface are discussed.

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1. Introduction

Zirconia holds an exclusive place amongst dental restorative materials compared with other oxide ceramics, such as alumina, because of its excellent mechanical properties as a consequence of transformation toughening that was identified in the mid-1970s [1]. Pure zirconia can exist in three different crystal structures depending on temperature. At room temperature up to 1170 °C, the symmetry is monoclinic, the structure is tetragonal between 1170 and 2370 °C and cubic above 2370 °C up to the melting point [2]. The transformation from tetragonal (t) to monoclinic (m) during a cooling process is accompanied by a volume increase (approximately 4%) and shear distortion, sufficient to cause catastrophic failure. Alloying pure zirconia with stabilizing oxides such as Y₂O₃ allows the preservation of the meta-stable tetragonal structure at room tempera-

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ture and therefore the potential to enable stress-induced \( t \rightarrow m \) transformation, which can enhance resistance to crack extension leading to higher toughness compared to alumina [1,3,4].

As a consequence of the metastability of tetragonal zirconia, stress-generating surface treatments, such as grinding or sandblasting, are able to trigger the \( t \rightarrow m \) transformation with the associated volume increase leading to the formation of surface compressive stresses, thereby increasing the flexural strength. However such metastability of the material also increases the susceptibility to aging [5]. The low temperature degradation (LTD) of zirconia is a well-documented phenomenon, dependent upon the presence of moisture and modest heat [6-11]. The consequences of this aging process are multiple and include surface degradation with grain pullout and micro-cracking as well as strength degradation. Although LTD has been shown to be associated with a series of orthopedic hip prostheses failures in 2001 and despite a well established definition of the conditions under which LTD occurs, there is currently no clear relationship between LTD and failure predictability when zirconia is used as a dental bio-ceramic [12].

3Y-TZP is now widely used in dentistry for the fabrication of dental restorations, mostly processed by machining of partially sintered blanks followed by sintering at high temperature. The mechanical properties of 3Y-TZP strongly depend on its grain size [13-15]. Above a critical size, Y-TZP is less stable and more vulnerable to spontaneous \( t \rightarrow m \) transformation than smaller grain sizes (<1 \( \mu \)m) [16]. Moreover, below a certain grain size (~0.2 \( \mu \)m), the stress-induced transformation is not possible, leading to reduced fracture toughness [17].

Porcelain fused to metal (PFM) restorations developed over decades have placed great attention on the preparation of the interface prior to building up of the porcelain as well as the reactions that promote adhesion. The nature of the interface between Y-TZP and its veneering porcelains however has not been carefully studied. Although diffusion processes as the reactions that promote adhesion. The nature of the interface between Y-TZP and its veneering porcelains however has not been carefully studied. Although diffusion processes are time-dependent, chemical reactions may occur between the two ceramic materials [15,18]. In a previous paper the authors used high resolution SEM to investigate the surface features of Y-TZP bonded to porcelain and found that wet thick layers of porcelain when sintered generated highly textured Y-TZP grains [18]. The aim of this paper is to use X-ray micro-diffraction to evaluate the interface region between veneering porcelain and Y-TZP prepared using standard dental fabrication techniques.

### 2. Materials and methods

In this study yttria partially stabilized tetragonal zirconia polycrystalline ceramic (Y-TZP, VITA In-Ceram® YZ, VITA Zahnfabrik, Germany) is used as a framework material. This material was prepared by sintering blocks, suitable for CAD/CAM machines Cerec® and InLab® (Sirona, USA), at a temperature of 1530 °C for 2 h. For the observation of the interface system rectangular plates with dimensions of 10 mm × 10 mm × 1 mm were used. As veneering porcelain, VITA VM® 9 (colour shade 2M2), prepared in three variations were built up on the zirconia surface using the methods listed in Table 1.

Prior to veneering the samples the flat zirconia surface was evaluated by X-ray diffractometry to determine the crystal phases of the sintered zirconia grains. This step ensured that changes at the zirconia interface following porcelain build up were not associated with transformations due to exposure to room temperature humidity prior to porcelain veneering.

To assess the initial wetting of the Y-TZP a so-called Wash-Dentin-firing procedure was used. This involves the firing of a very thin layer of veneering porcelain (approximately 0.05 mm thick) to 950 °C at a heating rate of 45 °C/min with a holding time of 2 min, following a preheating temperature at 500 °C for 6 min in a 70% closed furnace chamber. The same Wash-Dentin firing temperature cycle was used for each preparation method. For all preparation methods, after preheating at the holding period for 2 min, vacuum was applied upon closing of the furnace chamber before heating to the highest temperature. For sample method number 2, a thicker moist layer (approximately 2 mm thick) of veneering porcelain was applied prior to initial heating. To observe only the initial bonding reaction between the two different materials no further firing process was conducted and no other porcelain layer was applied. The liquid application medium for the porcelain was systematically varied from complete absence in sample method 3 of the standard proprietary wetting liquid (VITA VM Modelling Liquid, VITA Zahnfabrik, Germany) while for samples prepared according to method number 1 and 2 this was included. The choice of the standard liquid was based upon observations in the previous study that faceting occurred in the presence of both distilled water and the VITA VM liquid [18]. The furnace used during this study was a VITA Vacuum 4000 (VITA Zahnfabrik, Germany).

To enable a magnified view of the interface, taper sections at approximately 5° to the interface of all samples were cut with a water-cooled diamond saw (600 microns Struers, Germany) and polished with diamond paste (3 \( \mu \)m VITA Zahnfabrik, Germany). The cross-sections of the samples were examined by scanning electron microscopy (LEO 438 VPI Carl Zeiss SMT and LEO 1530 Gemini, field emission microscope) and an XRD2 micro-diffractometer (BRUKER-D8 DISCOVER with General Area Diffraction Detection System (GADDS) equipped with a specially developed focusing micro-lens to generate a analysing spot of approximately 50 \( \mu \)m diameter (FWHM) [19]).

Traditionally X-ray diffractometers used for the purpose of local area analysis are typically equipped with pinhole

### Table 1 – Preparation methods of the Y-TZP frameworks.

<table>
<thead>
<tr>
<th>No.</th>
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<th>Firing process</th>
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<tr>
<td>1.</td>
<td>VITA VM liquid</td>
<td>VITA VM9 Wash-firing (950 °C)</td>
</tr>
<tr>
<td>2.</td>
<td>VITA VM liquid</td>
<td>VITA VM9 Wash-firing thicker layer (950 °C)</td>
</tr>
<tr>
<td>3.</td>
<td>No</td>
<td>VITA VM9 Wash-firing (950 °C)</td>
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collimators to generate a small analysis spot on the sample surfaces. The use of such pinhole collimators however decreases the intensity of the X-ray beam on the sample dramatically, which results in long measurement times. In the last decade pinhole collimators have been replaced by monocapillary optics and in the last few years by focussing polycapillary micro-lenses in order to focus the generated X-ray beam on the sample. This has increased the localized intensity on the sample by orders of magnitude depending on the X-ray source and type of optics in comparison to a standard pinhole collimator.

The X-ray micro-diffractometer (μ-XRD²) used in this study was a modification of typical powder diffractometer with a focussing micro-lens to achieve a micrometer-sized X-ray beam and a 2-dimensional detector system (BRUKER-HiStar), which covers app. 30° in 2θ and app. 30° ϕ at the same time. The advantage of such a focussing polycapillary micro-lens instead of the common pinhole collimator or a monocapillary is the short measurement time required, down to a few seconds, due to the high brilliance of the X-ray beam on the sample combined with a spot size currently down to approximately 50 μm diameter FWHM. A general disadvantage of small spot sizes in powder diffraction setups is the potentially poor crystallite statistics in the analyzed volume depending on the crystallite size. In addition and to avoid this disadvantage the additional 2-dimensional HiStar-detector provides direct assessment of texture effects and crystallite size in the analyzed sample.

Before commencing the μ-XRD²-analysis of the interface region various sample preparation methods of the taper sections were examined by locally scanning the surface with the micro-diffractometer. This indicated that the cutting and polishing preparation did not generate significant t → m transformation of the zirconia grains on the surface. Monoclinic peaks and broadening of the tetragonal/cubic peak were only observed on the ground surface of the prepared area and not on the polished area in the vicinity of the veneered interface with the relatively low spatial resolution of the X-ray diffraction system used in the former study.

All three preparation procedures were scanned with the micro-diffractometer in the manner shown in Fig. 1. The veneered part and the polished zirconia surfaces were measured at 8 different positions in steps of 50 μm across the interface with a measurement time of 120 s per pattern using the 50 μm micro-lens and Co Kα (radiation with 30 kV/30 mA setting) with a fixed incident angle of 10° to the flat interface. Due to this incident angle the measurement spot has an elliptical geometry with a length of app. 400 μm and a width of app. 50 μm. Three of the scanning positions are shown as an example for the working area in Fig. 1 where the middle position focuses on the location directly at the interface between Y-TZP and porcelain and the others on the adjacent positions. The schematic on the left hand side vertically through the tapered interface sample shows positions of the spots indicated by the elliptical areas on the right hand side. No etching process such as HF content gel was used to ensure that other potential influences on the framework material were minimized.

3. Results

The zirconia surfaces were scanned, as described above, before the veneering process to ensure that the zirconia was completely tetragonal and had no observable monoclinic peak.

The GADDS frame in Fig. 2 shows only the tetragonal crystal structure on the surface of the Y-TZP sample. In all samples reflections no monoclinic Baddeleyite structure of ZrO₂ was observed for untreated Y-TZP with a solid angle of incidence of 10°. The major peak is associated with the 1 0 0 tetragonal/cubic peaks while the less intense peaks to the right are of the 2 2 0 series of tetragonal and cubic peaks.
Fig. 3 – X-ray scans across the interface area of the Wash-Dentin (method 1) prepared sample measured using a 50 μm micro-lens, Co Kα, 30 kV/30 mA, fixed incident angle 10°, 120 s/frame.

Fig. 4 – X-ray scans across the interface area of the thin layer of porcelain coated zirconia (preparation procedure 1) measured using a 50 μm micro-lens, Co Kα, 30 kV/30 mA, fixed incident angle 10°, 120 s/frame. An arrow at position 2 correlates the intense single crystal reflection with the corresponding monoclinic (−1 1 1)-reflection in the XRD-pattern.
detectable. After the phase composition of the Y-TZP surfaces was established they were veneered using the different sample preparation methods listed in Table 1.

The result of the μ-XRD²-measurements from one typical sample prepared with a Wash-Dentin (preparation method no. 1) coating and prepared as schematically shown in Fig. 1 are presented in Fig. 3. The black arrow on the left side at position 1 identifies the location of the interface of the taper section between the veneered zirconia grain surface and the polished zirconia. Scans of intensity versus 2θ at 8 measurement points were performed, starting in the veneered surface, proceeding across the interface region and ending in the substrate zirconia.

Both measurements at position 1 (green coloured patterns) show only low intensities of both, monoclinic and tetragonal/cubic Zirconia due to the overlying veneering glass, which absorbs the X-ray beam.

At the locations 2 and 3 (red patterns) the intensities from the zirconia surface beneath the porcelain layer were significantly increasing, due to the decreasing thickness of the overlying veneering. It should be pointed out that the observed monoclinic (1 1 1)-reflection which appears at app. 33° 2θ seems to be significantly more intense than for the other locations. Also in both green patterns (positions 1 and 2) there is a significantly intense monoclinic reflection at this position.

At positions 4–7 (black patterns) no porcelain is covering the zirconia substrate due to the geometry of the sample, therefore the information comes from the ground and polished zirconia at areas below the interface. These areas are beneath the veneered zirconia interface and so they provide a basis for comparison with the diffraction patterns near the interface. These results are similar to positions 2 and 3 (red patterns). The X-ray patterns show broader tetragonal/cubic peaks and weak and significantly broad monoclinic peaks at 2θ 33° in all of these areas. As such the influence of the veneering
Fig. 7 – Interface area of the without liquid veneered zirconia (preparation procedure 3) measured using a 50 μm micro-lens, Co-Kα, 30 kV/30 mA, fixed incident angle 10°, 120 s/frame, only diffraction rings caused by the tetragonal ZrO₂ are observable.

process can be compared with the grinding/polishing preparation of the taper section.

The right hand side of Fig. 3 presents the scan from 2θ 32° to 37° at a higher magnification superimposing results from all locations. The slight broadening in width of the known cubic/tetragonal peak at about 2θ 35° is evident for the black traces and an increase in intensity and sharpness of the monoclinic peak at 2θ 33° for some of the green and red traces is also obvious.

Another feature evident in Fig. 3 is the two reflections in the range around 70° 2θ, the cubic (3 1 1)/tetragonal (0 1 3 and 1 2 1) peaks. Both show a switch of tetragonal peaks intensity (1 2 1 to 0 1 3), starting under the veneering (red patterns 2, 3). This grinding induced intensity flip phenomenon of these tetragonal intensities is well known on zirconia surfaces [20].

The μ-XRD²-results for the “normal” Wash-Dentin layering technique (preparation method no. 1) coating and the thicker veneered specimen (preparation method no. 2) show similar results. It was impossible with the diffraction method to recognize dissimilar effects between the zirconia samples from these two preparation methods as anticipated from other studies [18]. In both cases clear monoclinic zirconia peaks were detectable directly under the porcelain layer from the zirconia surface, but they were not stronger or more clearly recognizable from those associated with the preparation with the thicker layer (method no. 2) than the layering technique (method no. 1).

To understand the reason for the sharp and increasing (−1 1 1)-reflection of the monoclinic phase especially in the interface region in Fig. 3 the related GADDS-frames from the Hi-Star detector are shown in Fig. 4 for the 6 patterns which are in the direct neighbourhood of the interface region.

The GADDS frame especially for position 2 shows a very strong single crystal reflection which is the reason for the monoclinic (−1 1 1) reflection in the corresponding pattern (The arrow indicates this single crystal diffraction spot within the corresponding reflection pattern).

Higher magnification X-ray patterns provide a better appreciation of the structural phase change with the Wash-Dentin coating and the thicker layer of the porcelain on top of Y-TZP (preparation no. 1 and no. 2). Figs. 5 and 6 present azimuthal projections from positions at similar distances from the interface region with preparation methods 1 and 2 within the veneered areas measured in this case with a 500 μm monom-capillary optic with a 300 μm exit pinhole to analyse a larger surface area for better statistics. Again in both cases similar single crystal reflection spots from the monoclinic phase are detectable in the area under the porcelain independent of the preparation method.

A clearly identifiable difference in the Y-TZP surface region prepared in the absence of a liquid medium by the veneered
process (preparation method no. 3, Fig. 7) was the absence of a detectable monoclinic peak through the porcelain very close to the interface.

4. Discussion

The present results indicate that prior to the veneering procedures on the surface of the zirconia samples only a tetragonal/cubic crystalline structure is detectable. After two different sample preparation methods 1 and 2, with either a thick wet porcelain layer or a thin wash coat layer with the presence of modelling liquid, the monoclinic phase was detectable by micro-diffraction beneath the porcelain layer. Furthermore in the polished and ground areas of the unveneered area, a noticeable increase in the width of the tetragonal/cubic peak intensity and a small broad monoclinic peak was visible. The observation was similar to what Coldea et al. [21] and Denry et al. [22] observed following different grinding and polishing procedures on Y-TZP-surfaces. The difference here is that the same observations were made after veneering on a zirconia surface, which was not affected by any grinding procedure.

These clearly observable features appearing after the veneering process point to crystal changes that develop on the Y-TZP grains directly at the zirconia-porcelain interface from $t\rightarrow m$ transformation as a consequence of the veneering. It indicates that a significant structural change has developed during the veneering process with the presence of a liquid medium during furnace firing. There is clear evidence of broadening of the tetragonal/cubic zirconia diffraction peak accompanying a limited rise of the monoclinic (1 1 1) intensity after firing the damp porcelain (preparation method no. 1, Fig. 3 and no. 2, Fig. 6).

An approximate estimate of the extent of the monoclinic present in the diffraction patterns shown in Figs. 3, 6 and 7 for the different preparation methods may be obtained using the simple analysis proposed by Garvie and Nicholson [3]. It utilizes the intensities of the monoclinic and tetragonal/cubic diffraction peaks. The results are given in Table 2 below.

These results indicate the in the presence of liquid (methods 1 and 2) the monoclinic content from the surface (position 2) is far greater than for the same position in the absence of liquid (method 3) and also greater than the extent of monoclinic generated by cutting, grinding and polishing of the zirconia (position 5) for all methods below the interface. But due to a more detailed and critical examination of the GAADS frame from the 2-dimensional Hi-Star detector shown in Fig. 4 especially at Pos. 2, it is important to point out, that the monoclinic information is caused by one single crystal reflection in contrast to the tetragonal/cubic reflection which shows complete diffraction rings typical for a random crystal orientation of this phase. For this reason an analysis like that proposed by Garvie and Nicholson [3] of these samples is not entirely appropriate. However even without a quantitative measurement, the samples consisting only of the porcelain powder and no liquid medium exhibited far less observable monoclinic phase than the other two preparation methods. These results are consistent with other observations that humidity at elevated temperatures causes the $t\rightarrow m$ nucleation and growth [18,23].

A significant feature of the 2-dimensional HiStar-detector associated with the $\mu$-XRD$^2$-setup is that from the intensity distribution along the diffraction ring of each reflection, direct information of crystallite size and texture could be estimated. In the case of the tetragonal/cubic zirconia crystals present the intensity distribution is completely uniform which indicates that the crystallites are statistically randomly distributed in the measurement volume. However for the monoclinic phase only isolated peaks as indicated with arrows in Figs. 4–6 are observed. This suggests the monoclinic crystall could present are highly aligned. This arises because the nucleation of the tetragonal to monoclinic transformation at a surface will be governed by both the strain energy constraints and also specific orientation requirements between the tetragonal and monoclinic phases [23]. These authors also show that at a surface transformation of the tetragonal to monoclinic can only occur by one lattice correspondence. That is, certain oriented grains will be more susceptible to this transformation and result in highly textured or oriented monoclinic volumes, which is what the GAADS images indicate. In addition the highly oriented X-ray beam will show the presence of these diffraction peaks at localized spots as observed.

The use of only porcelain powder (preparation method no. 3) without any liquid medium resulted in no obviously detectable transformation of the zirconia grains structure, indicating that the powder itself, even with the same temperature exposure as with the other preparation methods, cannot be the reason for developing a monoclinic peak from the Y-TZP grains. In contrast, the changes observed using the liquid medium with exactly the same porcelain powder and the same firing procedure clearly shows an observable effect. These observations support previous studies [10,18] on the role of humidity during the initial stages of the moist porcelain powder heating up leading to the destabilization or LTD of the veneered Y-TZP tetragonal grains. This behaviour is in agreement with the well-documented occurrence of instability of Y-TZP in the presence of humidity [5,10,12,18]. The present results for the Y-TZP grains are similar to the initial stages of moisture induced tetragonal to monoclinic phase transformation observed for these materials using atomic force and optical microscopy by Chevalier [5,10,12]. In addition Chevalier et al. were unable to detect the presence of monoclinic phase using X-ray diffraction as the layer thickness and amount of tetragonal transformed to the monoclinic phase was very limited. The X-ray micro-diffraction setup used in this case study with the 10° incident angle using a focussing micro-lens to

<table>
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<th>Monoclinic V% @ black line position 5</th>
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<tbody>
<tr>
<td>Method 1 (wash-firing)</td>
<td>11.5%</td>
<td>3.5%</td>
</tr>
<tr>
<td>Method 2 (thick layer)</td>
<td>13%</td>
<td>4%</td>
</tr>
<tr>
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The present results indicate that prior to the veneering procedures on the surface of the zirconia samples only a tetragonal/cubic crystalline structure is detectable. After two different sample preparation methods 1 and 2, with either a thick wet porcelain layer or a thin wash coat layer with the presence of modelling liquid, the monoclinic phase was detectable by micro-diffraction beneath the porcelain layer. Furthermore in the polished and ground areas of the unveneered area, a noticeable increase in the width of the tetragonal/cubic peak intensity and a small broad monoclinic peak was visible. The observation was similar to what Coldea et al. [21] and Denry et al. [22] observed following different grinding and polishing procedures on Y-TZP-surfaces. The difference here is that the same observations were made after veneering on a zirconia surface, which was not affected by any grinding procedure.

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achieve a 50 by 200 μm-sized localized beam had the advantage to enable phase stability of the near surface layer of the zirconia interface to be evaluated. This specially designed X-ray polycapillary micro-lens system also enabled detection of the zirconia through thin layers of the porcelain surface.

This structural t → m change at the interface creates localized residual stresses as a consequence of the volume dilation as well as the different coefficient of thermal expansion of the framework material. Such localized residual stresses at the interface may weaken the porcelain to zirconia adhesion and may have influenced the shear interfacial strength tests by Fischer et al. [24]. However to date there have been no published reports of clinical adhesion failure between porcelain and Y-TZP frameworks despite increasing observations of chipping induced failures within the porcelain on such frameworks [25].

The localized stresses developed between the veneering and the reliability of the dental restoration in the clinical situation are not completely established and further investigations with a focus on these topics need to be undertaken.

5. Conclusions

The present observations using the locally resolved μXRD²-technique have clearly established that the porcelain veneering process, especially with a wet veneer during firing, results in a localized tetragonal to monoclinic structural transformation at the surface of the zirconia framework material during preparation of these all-ceramic dental restorations.

In the case of moist veneering porcelain a highly textured or oriented monoclinic crystalline phase was observed at the zirconia/porcelain interface. In the absence of moisture within the veneering porcelain no transformation of the Y-TZP tetragonal phase was identified.

As a consequence of the findings in this study, it is strongly recommended to use a porcelain layering technique that is very thin and as dry as possible for the initial layering application to prevent destabilization of the tetragonal crystals of the Y-TZP framework at the interface, which will otherwise induce local mechanical stress into the overlying porcelain layer and therefore could decrease the mechanical stability of the final product.

REFERENCES