

Zirconia in dentistry - understanding the effects of yttria-content, surface processing and heat-treatment on surface and mechanical properties

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ABSTRACT

Zirconia materials with 3–5 mol% of yttria are widely used in dentistry for implants, abutments, crowns and fixed dental prostheses depending on the required aesthetics and mechanical reliability. Nevertheless, in-depth analysis of the underlying mechanisms affecting the surface and mechanical properties of dental zirconia is lacking to date. The aim of this study was therefore to understand the effects of yttria-content, surface processing and heat-treatment on surface and mechanical properties. Five different zirconia materials varying in yttria-content with 3 different surface processing (as-sintered, polished, polished and heat-treatment) were therefore systematically investigated. Of these materials, morphological properties such as grain sizes and surface roughness were characterized, crystal phase composition of monoclinic, tetragonal, or cubic phase assessed, biaxial flexural strength and resistance to scratching evaluated. The study highlights that surface and mechanical properties of zirconia are determined by the yttria-content, surface processing and resulting crystallographic composition, presence of grain boundaries and grain sizes on the surface, as well as surface roughness in general. By polishing and heat-treatment of the zirconia specimens, it was possible to eliminate macro-structures and generate surfaces with distinct grain sizes in the mid nano-range while increasing strength significantly. The polymorphic behavior of zirconia is highly complex and not yet fully understood, mainly owned to interrelations between crude material properties and production processes that ultimately affect end-product material characteristics.

1. Introduction

The ongoing improvement of restorative materials in dentistry for implants, abutments, crowns and fixed dental prostheses focuses on ceramic materials that exhibit both natural aesthetics and high mechanical reliability. A frequently used ceramic is zirconia (ZrO_2), a polymorph material existing in different crystalline configurations, depending on stabilizing metal oxide dopants, stress, temperature, humidity or pressure [1–3]. The monoclinic phase of zirconia is stable between room temperature up to 1170 °C, while the tetragonal or cubic phase are only metastable at these temperatures [1,4]. In dentistry, dopants such as yttria (Y_2O_3) are added to zirconia to stabilize the

metastable crystal phases with an effect on the materials' strength [5]. The most frequently used material for dental implants and frameworks of fixed dental prostheses is a tetragonal zirconia polycrystal stabilized by adding 3 mol% of yttria (Y_2O_3), termed 3Y-TZP [6]. This material provides a high strength over 1000 MPa due to toughness enhancement mechanism by phase transformation. When subjected to mechanical [7, 8] or thermal [9–11] stress, the material can undergo a tetragonal to monoclinic phase transformation that is accompanied by a volumetric expansion of 4–5 % [12]. The resulting effect that emerging cracks are compressed and thus closed is termed "transformation toughening". Due to sandblasting or grinding of the surface, performed during processing of dental ceramics, the cubic or tetragonal phase may partially transform

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to a metastable hettotype of cubic phase with a 15 % volume increase [1, 13–15].

However, 3Y-TZP is less translucent and opalescent than human teeth as it is composed of grains in the submicron range, hence light is scattered at the large number of grain boundaries. For clinical applications, 3Y-TZP restorations are therefore covered with silicate ceramic to mimic the optical properties of natural teeth [3]. However, this veneering glass is prone to chipping, affecting around 2 % of crowns and 13 % of fixed dental prostheses after 5 years [4,5]. Hence, to eliminate the need for veneering glass, different approaches have been implemented to improve the translucency of zirconia ceramics. One of these is an increase in yttria-content to 4–6 mol%, causing the formation of larger grains and less grain boundaries with less light interaction due to the presence of significant fractions of cubic crystal phase, that is not providing a transformation toughening effect [7,16,17]. These materials are termed "partially stabilized zirconia" (e.g. 4-6Y-PSZ) [5].

Initial processing of zirconia for restorations is typically done by computer-aided design/computer-aided manufacturing (CAD/CAM) in dental laboratories. The restorations are digitally designed and then milled over-dimensionally to compensate sintering shrinkage out of commercially available blanks. These blanks are made from zirconia ready-to-press powder and pre-sintered to porous state to guarantee optimal milling. Blanks having varying yttria-content providing the required translucency and strength to the intended restoration are available [11]. The restorations are then sintered at specific temperatures from 1450 to 1550 °C to densify the zirconia, which is accompanied by a shrinking of about 20 % and subsequently polished or stained and glazed.

Fabrication of zirconia implants is done by the respective manufacturers using 3Y-TZP. In addition to sintering, the 3Y-TZP material is often additionally densified by hot isostatic pressing (HIP) [18]. As milling of zirconia implants is a rather sensitive and expensive working step, implant production by injection-molding is currently trending [19, 20]. Here, a zirconia feedstock is injected in a prefabricated negative implant mold, then slowly debinded, sintered and optionally HIPed.

The endosseous part of milled or injection-molded zirconia implants is then commonly micro-structured using sandblasting and etching [21], which creates a micro-rough surface, but also a significant fraction of monoclinic phase, making the implant susceptible to aging [22]. To again retrieve the tetragonal phase, the implants can be subjected to another heat-treatment at 1250 °C [22–25]. In summary, despite excellent previous research focusing on transformation toughening of zirconia and related investigations on material properties [1–4,7,13,18, 26], it remains unclear how distinct processing parameters affect end-product properties. Specifically, it is not fully understood how polishing and subsequent heat-treatment of zirconia with varying yttria-content and processing affect the crystal phase composition and mechanical performance. As such, the aim of this study was to gain an in-depth understanding of the interrelations between yttria-content,

surface processing and heat-treatment, and their correlations with surface and mechanical properties. This study highlights the importance of considering multiple parameters, including crude material composition and processing variables, on a large set of end-point characteristics such as the crystallographic composition, presence of grain boundaries, grain sizes on the surface, and resulting surface roughness. Knowledge over these parameters and their correlation will allow one to choose appropriate raw materials or manufacturing processes to gain control over specific parameters and address requirements for aesthetic solutions without compromising mechanical properties.

2. Materials and methods

2.1. Specimen production

To study the effects of yttria-content, surface processing and heat-treatment on chemical, crystallographic and mechanical properties of zirconia, disk-shaped specimens of 5 different materials were prepared. Two materials that are currently used for commercially available zirconia implants were processed by the respective manufacturers using injection-molding, debinding and sintering (3YniZ, Maxon Dental, Kenzingen, Germany) or grinding, sintering and HIPping (3YnZ, CeramTec, Plochingen, Germany) (Table 1). The materials were provided by the respective manufacturers in the form of as-sintered disks of $\varnothing$ 15.0 mm $\times$ 1.5 mm (3YniZ) or $\varnothing$ 12.7 mm $\times$ 2.0 mm (3YnZ). Disks of 3 materials varying in their yttria-content that are commercially available to fabricate dental restorations were additionally processed to disks of $\varnothing$ 12.7 mm $\times$ 2.0 mm (3YZ, 4YZ, 5YZ, Straumann, Basel, Switzerland). An STL file of a disk was therefore designed, positioned in the middle layer of the white state blank ($\varnothing$ 98.5 mm, 14 mm height), CNC-milled (PM7, Ivoclar, Schaan, Liechtenstein) and subsequently sintered (Vita Zyrcomat 6100 MS, Vita, Bad Säckingen, Germany) with a heating rate of 5 °C/min to 1500 °C for 120 min and a cooling rate of 10 °C/min to 800 °C. Materials varying in yttria content for restorations were chosen to gain an in-depth understanding of how grain size influences material properties. Since restorative materials in the oral cavity are also in direct contact with soft tissue it is of relevance to also include restorative materials and distinguish their behavior from zirconia implant materials.

Twenty disks of each material were tested in the as-sintered state (machined group "m") (Fig. 1). The surface of 40 disks of each material was polished (group "p") (Tegamin-25, Struers, Ballerup, Denmark) using an automated protocol (Piano 220 diamond disc; MD-Plan disc and 9 μ m, 3 μ m, 1 μ m DiaPro suspension, Struers). To retrieve the grain structure on the surface, 20 specimens per group were heat-treated (tempered, group "t") using a heating rate of 17 °C/min to 1250 °C for 60 min and a constant cooling rate of 17 °C/min to 200 °C (Vita Zyrcomat 6100 MS, Vita) [24].

Table 1
Materials used and characteristics as provided by the respective manufacturers.

Code	Material, Manufacturer	Composition	Flexural strength	Application
3YniZ	TZ-3YS-E, Maxon Dental, Kenzingen, Germany	ZrO ₂ , 5.15 wt% Y ₂ O ₃ , 0.25 wt% Al ₂ O ₃ , <3.0 wt% HfO ₂	1500 MPa	Injection-molded zirconia implants
3YnZ	MZ111, CeramTec, Plochingen, Germany	ZrO ₂ , 5.0 wt% Y ₂ O ₃ , 0.1 wt% Al ₂ O ₃ , 1.9 wt% HfO ₂	1471 MPa	Zirconia implants
3YZ	n!ce Zirconia LT, Straumann, Basel, Switzerland	88.0–95.0 wt% ZrO ₂ , 4.0–6.0 wt% Y ₂ O ₃ , $\leq$ 0.5 wt% Al ₂ O ₃ , $\leq$ 5.0 wt% HfO ₂	>1200 MPa	Framework fixed-dental prostheses
4YZ	n!ce Zirconia HT, Straumann, Basel, Switzerland	87.0–95.0 wt% ZrO ₂ , 5.0–8.0 wt% Y ₂ O ₃ , $\leq$ 0.5 wt% Al ₂ O ₃ , $\leq$ 5.0 wt% HfO ₂	>1200 MPa	Monolithic fixed-dental prostheses
5YZ	n!ce Zirconia XT, Straumann, Basel, Switzerland	85.0–93.0 wt% ZrO ₂ , 7.0–10.0 wt% Y ₂ O ₃ , $\leq$ 0.5 wt% Al ₂ O ₃ , $\leq$ 5.0 wt% HfO ₂	>600 MPa	Monolithic crowns and 3-unit fixed-dental prostheses

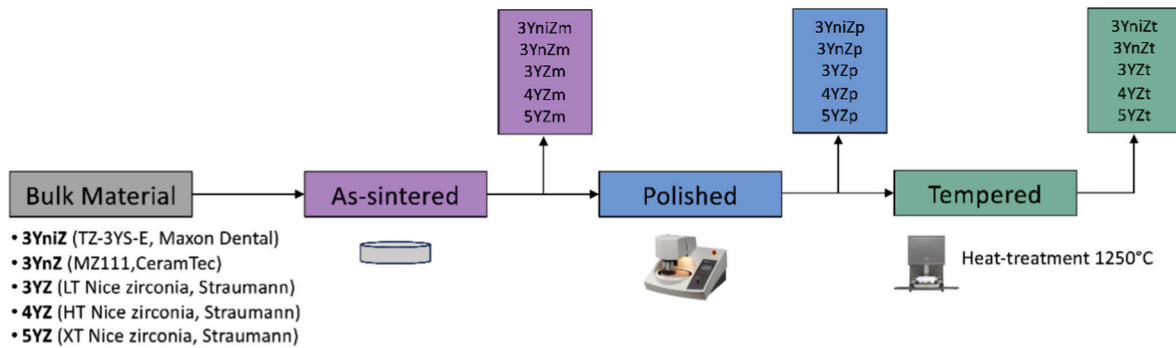


Fig. 1. – Experimental outline to produce zirconia specimens. Disk shaped specimens of 5 different raw materials (m) underwent surface polishing (p) or subsequent heat-treatment (t) (Total number of discs $N = 150$, $n = 20$ per experimental group).

2.2. Surface characterization

2.2.1. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX)

To visualize surface morphology and for elemental analysis, SEM was used (XL30 FEG, Philips, Eindhoven, The Netherlands). Acceleration voltage was set to 20 kV, using spot size 2, and working distance of 12 mm. One specimen per group was coated with a 20 nm thick gold layer (EM ACE600, Leica, Wetzlar, Germany) and images acquired at 3 different sites at a magnification of 10'000x and 20'000x. The zirconia grain size at the surface was determined for groups (m) and (t) using the 2D-linear intercept method [27].

Elemental composition of each material was additionally obtained using EDX (EDAX Apollo detector, Ametek, Berwyn, PA, USA) and EDX-software (Genesis Spectrum, version 6.53, EDAX, Mahwah, NJ, USA) at 20 kV, spot size 3 and 5'000x magnification. Subsequently, oxide compositions of ZrO_2 , Y_2O_3 , Al_2O_3 and HfO_2 were calculated and compared to manufacturer's specifications given in Table 1.

2.2.2. Surface roughness

The surface roughness parameter S_a (arithmetical mean height) was acquired using a laser scanning microscope (Sensofar S Neox, Sensofar, Barcelona, Spain). Data were obtained in confocal mode on 5 areas measuring $700 \mu m \times 850 \mu m$ on 3 specimens per group (20x objective, aperture 0.45, Nikon, Tokyo, Japan). Subsequently, the following filters were applied: denoising, waviness S-F Gaussian $2.07 \mu m$, roughness S-L Gaussian $30 \mu m$ and a polynomial degree 3 (SensoMAP Premium Software, Sensofar, version 8.2.10044, Barcelona, Spain).

2.2.3. X-ray diffraction (XRD)

The crystal phases of one sample per group were determined using X-ray diffraction (Rigaku MiniFlex 600C diffractometer, Rigaku Holdings Corporation, Akishima-shi, Tokyo, Japan) with $CuK\alpha$ -radiation at $1.54 \text{ \AA}$ (40 kV, 15 mA) from 20° to 90° (2θ), at a step size of 0.02° and scan speed of 1.0° per minute. The measurements were carried out using a sample turntable, with the rotations per minute (RPM) set to 20. Phase analysis was conducted using Rietveld refinement (Smartlab Studio II, Rigaku Holdings Corporation, Akishima-shi, Tokyo, Japan/version 5.0.174.0).

2.3. Mechanical properties

2.3.1. Flexural strength

Biaxial flexural strength was determined using the piston-on-three-balls test (Z020 Zwick/Roell, Ulm, Germany) according to ISO 6872 ($n = 15$ per group) [28]. Diameter and height of each specimen was measured beforehand with a digital caliper (Tesa technology, Tesa-Cal IP 67), specimens were placed with the treated side facing the three balls and testing was subsequently performed with a crosshead speed of

1.5 mm/min at room temperature. The load at fracture was measured and the biaxial flexural strength (σ) was calculated using equation (1):

$$\sigma = -0.2387 P(X - Y)/b^2 \quad (1)$$

where

σ : Biaxial flexural strength [MPa]

P: Load at fracture [N]

b: Thickness of the specimen at the fracture point [mm]

$$X = (1 + \nu) \ln(r_2/r_3)^2 + [(1 - \nu)/2](r_2/r_3)^2$$

$$Y = (1 + \nu) \left[1 + \ln(r_1/r_3)^2 \right] + (1 - \nu)(r_1/r_3)^2$$

where

ν : Poisson's ratio (0.25)

r_1 : Radius of the support circle [mm]

r_2 : Radius of the loaded area [mm]

r_3 : Radius of the specimen [mm]

2.3.2. Scratch test

Scratch tests were conducted using a tribometer (UMT TriboLab, Bruker, Billerica, Massachusetts, USA) equipped with a Rockwell diamond indenter (120°), and the corresponding analysis software (UMT TriboLab, Bruker, Billerica, Massachusetts, USA, Version: 1.143.275). The applied vertical normal force F_z and the lateral scratch force F_x were recorded 3 times on 3 specimens per group. The applied vertical force F_z was set such that it increased linearly from 4 N to 130 N over a total distance of 3 mm over 60 s. The scratch-induced damage on the specimens' surface was observed using SEM.

2.4. Statistical analysis

Data of grain size and surface roughness and biaxial flexural strength were tested for normal distribution using Shapiro-Wilk test and heteroscedasticity was plotted to display variances. For normally distributed grain size, surface roughness and biaxial flexural strength data, two-way ANOVA with Bonferroni post-hoc test was applied. The level of significance was set to $\alpha = 0.05$. In addition, Weibull two-parameter

Table 2

Oxide composition (mean $\pm$ standard deviation) calculated based on elemental analysis using energy-dispersive X-ray spectroscopy (EDX) on as-sintered (m) zirconia materials 3YniZ, 3YnZ, 3YZ, 4YZ and 5YZ ($n = 3$).

(mol%)	3YniZ	3YnZ	3YZ	4YZ	5YZ
ZrO ₂	93.3 $\pm$ 0.2	93.5 $\pm$ 0.1	93.2 $\pm$ 0.4	92.2 $\pm$ 0.3	90.9 $\pm$ 0.2
Y ₂ O ₃	3.9 $\pm$ 0.1	3.6 $\pm$ 0.1	3.9 $\pm$ 0.2	4.9 $\pm$ 0.1	6.3 $\pm$ 0.0
Al ₂ O ₃	0.4 $\pm$ 0.1	0.4 $\pm$ 0.0	0.6 $\pm$ 0.2	0.3 $\pm$ 0.4	0.4 $\pm$ 0.1
HfO ₂	2.3 $\pm$ 0.0	2.5 $\pm$ 0.0	2.3 $\pm$ 0.2	2.6 $\pm$ 0.1	2.4 $\pm$ 0.1

distribution of biaxial flexural strength data was plotted according to ISO 6872 [28] using the required minimum of 15 measurements per group due to limited material availability and Weibull modulus as well as Weibull characteristic strength were calculated.

3. Results

3.1. Surface characterization

The oxide composition of ZrO_2 , Y_2O_3 , Al_2O_3 and HfO_2 of the 5 different materials is given in Table 2. The materials mainly differed in Y_2O_3 content with lowest values found for 3YnZ (3.6 mol%) and 3.9 mol % for 3YnIZ and 3YZ. The Y_2O_3 content was increased for 4YZ (4.9 mol %) and 5YZ (6.3 mol%).

SEM images displaying the as-sintered (m), polished (p) or heat-treated (t) surfaces of the 5 different zirconia materials are shown in Fig. 2A. Grain formation is visible on surfaces (m) with smallest grains but also some small holes for 3YnZm, while largest grains are observed on 5YZ. Due to the polishing of the surfaces (p), grain structures are removed and grinding grooves become visible. After heat-treatment (t), the grain structure starts to re-appear, but not as pronounced as on as-sintered surfaces. Based on SEM images, grain sizes were quantified as displayed in Fig. 2B. Significant differences in grain size were found between materials ($p < 0.001$), but not for the surface treatment ($p = 0.565$). For material 3YnZ that also has the lowest yttria-content of 3.6 mol%, nano-sized grains of 167 ± 29 nm (m) and 190 ± 28 nm (t) were

measured. For 3YnIZ and 3YZ despite the similar yttria-content of 3.9 mol%, grain sizes varied. The initial grain sizes were retrieved after heat-treatment for all materials, except for 5YZ, where grain size decreased from 2310 ± 681 nm (m) to 1532 ± 328 nm (t). Additional data of all experiments is given in Supplemental Table S1.

The surface roughness parameter S_a indicating the arithmetical mean height was significantly influenced by the material ($p < 0.001$) and the surface treatment ($p < 0.001$) (Fig. 2C). Surface 3YnIZm revealed the highest surface roughness (333 ± 30 nm) reflecting the surface structure of the injection mold or the higher binder content of the feedstock of 16 wt% compared to the blank materials having roughly 4 wt% of binder. As 3YZm (165 ± 24 nm), 4YZm (174 ± 19 nm) and 5YZm (164 ± 36 nm) were processed with the same CNC milling machine, they also displayed similar S_a values due to the grinding process done with the same diamond instruments. Polishing reduced S_a irrespective of the material and the heat-treatment did not affect the surface roughness further within the measurement prospect of the confocal laser scanning microscope.

The crystal phase composition within a depth up to $70 \mu\text{m}$ of one specimen per group is displayed in Fig. 3. ISO 13356 requires zirconia implant materials to have less than 20 wt% monoclinic phase [29]. A small amount of 4 wt% of monoclinic phase was present for 3YnIZm, that was completely transformed to cubic phase or removed by polishing (p). The milled implant material 3YnZm displayed 38 wt% of monoclinic phase given that these samples were already produced several years ago and stored at ambient conditions. However, polishing induced a phase

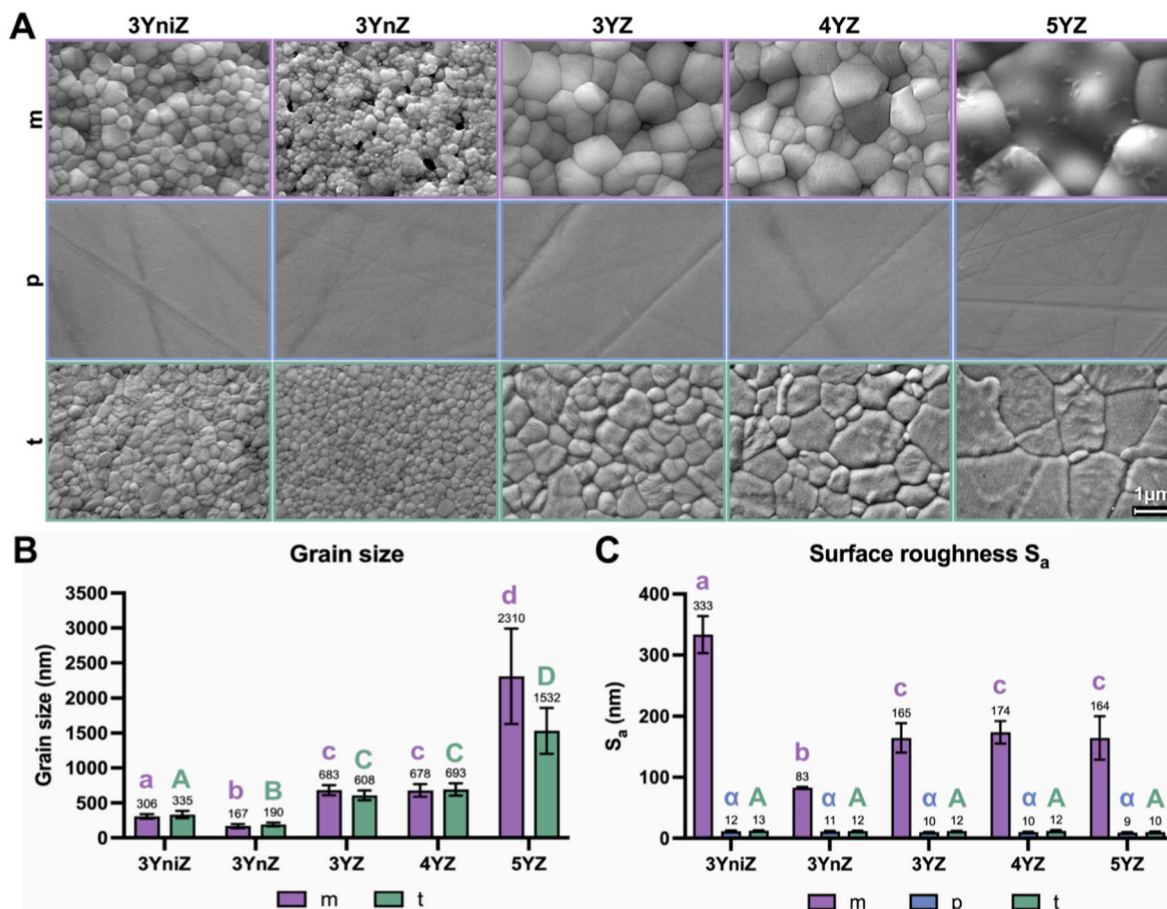


Fig. 2. (A) SEM images of as-sintered (m), polished (p) and tempered (t) surfaces of 5 different zirconia materials 3YnIZ, 3YnZ, 3YZ, 4YZ and 5YZ. Grain boundaries are visible on surfaces m and t, while on polished surfaces grinding grooves appear (B) Grain size mean and standard deviation ($n = 48$ measurements per group from 3 individual images). Statistical differences determined with Bonferroni ($p < 0.05$) between materials among groups m (lowercase letters) and t (uppercase letters) are indicated with varying letters. (C) Surface roughness parameter S_a indicating the mean arithmetical height ($n = 9$). Statistical differences determined with Bonferroni ($p < 0.05$) between materials among groups m (lowercase letters), p (Greek letters) and t (uppercase letters) are indicated with varying letters.

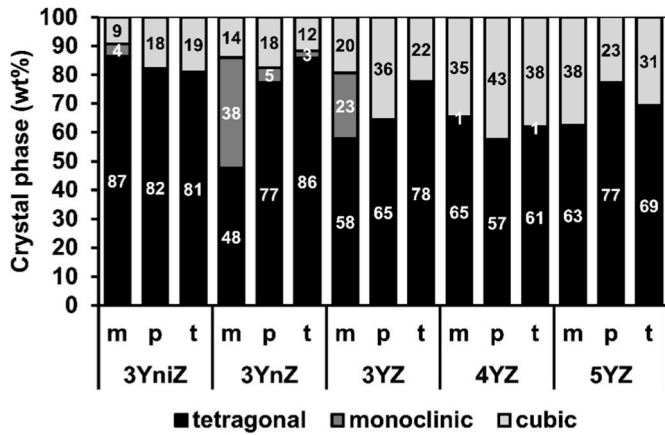


Fig. 3. Crystal phase composition in wt% on materials 3YniZ, 3YnZ, 3YZ, 4YZ and 5YZ of as-sintered (m), polished (p) and heat-treated (t) surfaces. Quantification is based on X-ray diffraction and refined by Rietveld with one specimen per group.

transformation to tetragonal from 48 wt% to 77 wt% or just manually removed the composition on the surface, while the tetragonal phase was further retrieved after heat-treatment to 86 wt%. For the restoration material 3YZ also 23 wt% of monoclinic phase was initially present that then transformed to cubic and tetragonal phase by polishing. The cubic phase was transformed to tetragonal phase by the heat-treatment. The 4YZ material was least affected by the surface processing, having around 57–65 wt% of tetragonal phase and 35–43 wt% of cubic phase. For 5YZ

tetragonal phase was increased from 63 to 77 wt% due to the polishing and the heat-treatment increased the cubic phase from 23 wt% to 31 wt%.

3.2. Mechanical properties

Biaxial flexural strength σ means and standard deviation as well as the Weibull distribution for each material are displayed in Fig. 4. The material ($p < 0.001$) and surface treatment ($p < 0.001$) affected flexural strength values significantly. For all materials, polishing significantly increased Weibull characteristic flexural strength as indicated by a shift to the right on the x-axis in the Weibull diagrams. However, polishing also reduced the reliability of materials 3YniZ, 3YnZ and 5YZ as observable by the lower slope of the trendline (Weibull modulus m). The subsequent heat-treatment significantly reduced the biaxial flexural strength again for 3YniZ ($p = 0.035$), 3YnZ ($p < 0.001$) and 3YZ ($p = 0.034$) but not for 4YZ ($p = 0.484$) and 5YZ ($p = 1$).

When exploring the scratch tracks on the surfaces using SEM, chipping was commonly found after 2.0 mm for 4YZ and 5YZ materials at around a normal force F_z of 88 N, irrespective of the surface treatment (Fig. 5).

3.3. Interactions of surface and mechanical properties

A correlation between grain size and yttria-content as well as biaxial flexural strength and yttria-content is described in Fig. 6. In Fig. 6A the grain size increases with an increased yttria-content. The correlation trendline for the heat-treated surfaces (t) is less steep as grain sizes for 5YZt are not fully recovered by the heat-treatment of 1250 °C for 60

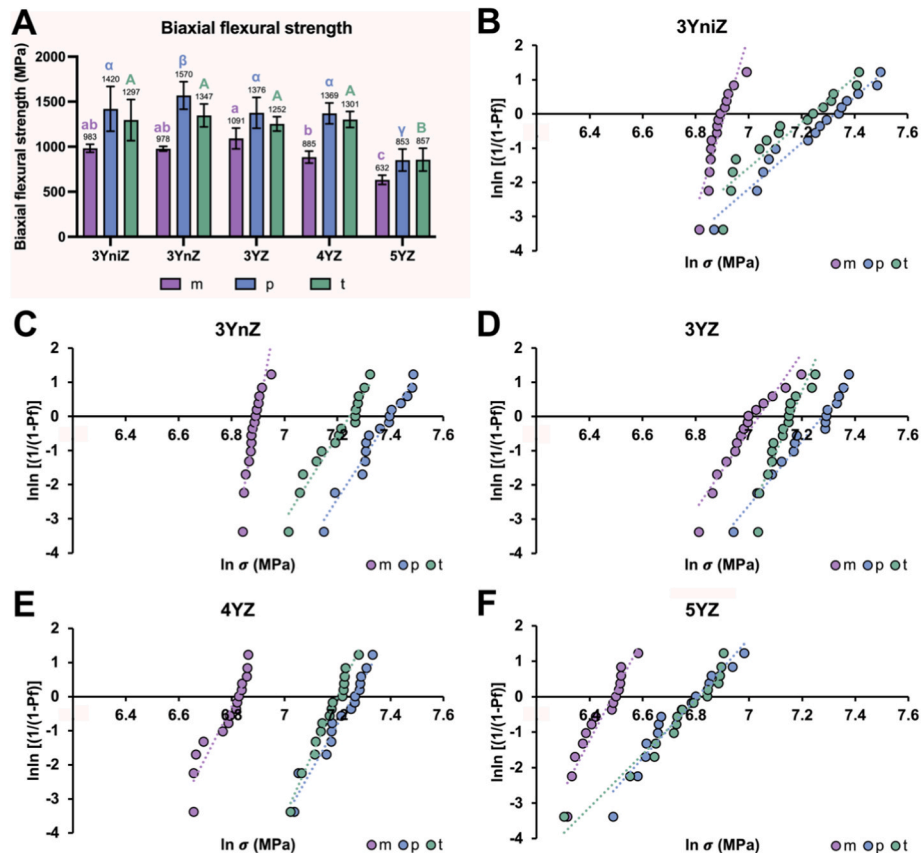


Fig. 4. (A) Biaxial flexural strength mean and standard deviations for all materials. Statistical differences determined with Bonferroni ($p < 0.05$) between materials among groups m (lowercase letters), p (Greek letters) and t (uppercase letters) are indicated with varying letters. The respective Weibull distributions of the five materials (B) 3YniZ (C) 3YnZ (D) 3YZ (E) 4YZ (F) 5YZ. A steep slope indicates a higher reliability of the material; a shift towards higher values on x-axis indicate a higher strength.

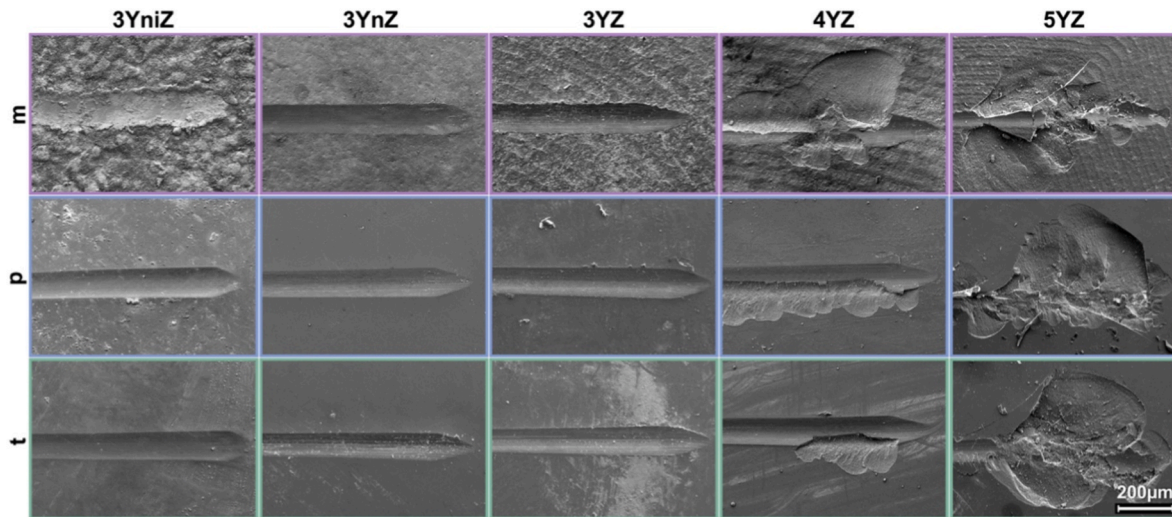


Fig. 5. Scanning electron microscope images acquired at the end of scratch track after a contact distance of 3 mm and final normal force F_z of 130 N. Chipping occurred for 4YZ and 5YZ, irrespective of the surface treatment.

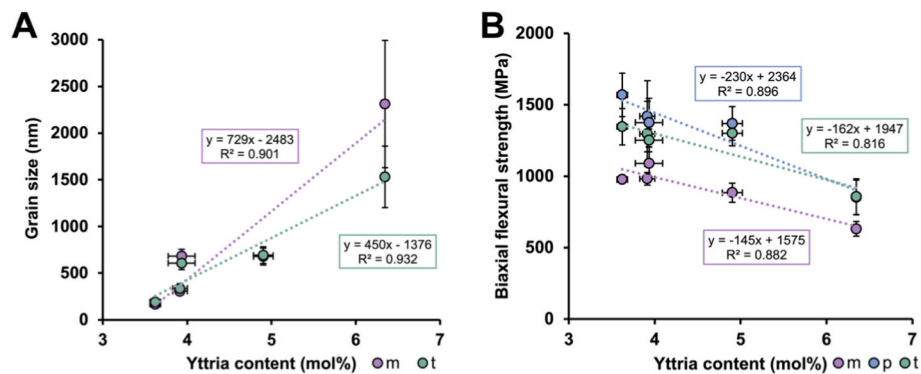


Fig. 6. (A) Linear correlation between grain size obtained in SEM images and yttria-content (EDX) of the 5 different zirconia materials after machining (m) and after heat-treatment (t). (B) Linear correlation between biaxial flexural strength σ and yttria-content (EDX) of the 5 different zirconia materials with as-sintered surfaces (m), polished (p) and tempered (t).

min. Fig. 6B shows that an increasing yttria-content results in a reduced biaxial flexural strength σ . In addition, the biaxial flexural strength σ is affected by the surface treatment and increases due to polishing and subsequent heat-treatment.

4. Discussion

The aim of this study was to gain an in-depth understanding of the effects of yttria-content, surface processing and heat-treatment on surface and mechanical properties of dental zirconia materials. Five zirconia materials that varied in their yttria-content with 3 different surface treatments were therefore analyzed. The measurements revealed that the polymorphic behavior of zirconia is highly complex and still not fully understood due to the interrelation between different parameters ultimately affecting the material properties. Nevertheless, important insights were highlighted throughout, and correlations were identified.

The tested zirconia materials presented a mol % yttria-content within the range defined by the manufacturers when converting EDX-derived wt% to mol%. Though, for all samples yttria values tended to be higher than common terminology for the respective materials. It needs to be acknowledged that EDX has an absolute measurement error of 0.1–3 wt%, and that the sputter gold coating of 20 nm thickness may have slightly affected the measurements by absorbing low-energy X-ray lines [30].

For the crystal phase composition, it was overall found that an

increasing yttria-content from 3 mol% to 5 mol% lowered the amount of tetragonal phase and increased the cubic fraction. The shape and size of the grains formed on the surface of the tested zirconia materials may have been subsequently affected by the presence of the different crystal structures. A higher amount of cubic phase due to an increased yttria-content resulted in overall larger grain formation within a range of 1532 ± 328 nm for 5YZ compared to 608 ± 70 nm for 3YZ. It is widely recognized that grains significantly larger than others and >1000 nm in SEM images are likely to be purely cubic grains [6].

The present findings imply that the resulting grain size on the surface is not only based on the crystal phases present in the respective material but is additionally affected by different raw materials, processing methods or sintering temperatures and as a result varying densification and grain growth. HIP treatment as it was done by the manufacturers for implant material 3YnZ was associated with lower grain sizes of 150–200 nm, while with the 3YZ material grain sizes in the range of 600–700 nm developed on the surface, potentially due to the higher sintering temperature. A previous study reported grain sizes ranging from 350 to 574 nm for different 3Y-TZP materials [31]. The heat-treatment as conducted for group (t) only affected the crystal grain size of 5YZ with grain sizes being around 800 nm smaller than on as-sintered surfaces of group (m). Enhanced grain growth at the surface is a typical phenomenon during the sintering of 5Y-TZP as observed on surfaces of group (m), primarily due to the diffusion-promoting and stress-free conditions in combination with the cubic phase content [16]. It was hypothesized,

that due to the polishing, these large grains were removed, and the smaller grain sizes of the bulk were exposed after heat-treatment for group (t).

In addition, grain size is associated with the aesthetic appearance of a dental zirconia material. The translucency increases when the grain size is increased by decreasing the grain boundary area, being considered as a source of light scattering [32–34]. Materials with a higher translucency as 4Y-PSZ or 5Y-PSZ are eligible for monolithic restorations without the need of veneering, hence, lowering costs. It has been shown that grain size can also be influenced by increasing the sintering temperature from 1250 °C to 1450 °C for 3Y-TZP materials, increasing grain sizes from 150 to 350 nm [35]. Hence, future studies should involve additional tempering regimes to on the one hand gain knowledge on the correlation between temperature and grain size formation, and on the other hand establish and validate manufacturing protocols to tune translucency by applied parameters to meet aesthetic demands for restorative zirconia materials.

Surface roughness of as-sintered discs (m) was impacted by material manufacturing and subsequent sample processing. The highest surface roughness value for the arithmetic mean height S_a of 333 nm was reported for the injection-molded material 3YnZ with the surface being structured by the feedstock material's fit to the mold topography or the feedstock itself due to its high binder content. S_a of CNC milled specimens of materials 3YZ, 4YZ and 5YZ was between 164 and 174 nm, being defined by the bur's milling strategy, diamond particle size of burs and pores of the pre-sintered blank material. For group m of 3YnZ, S_a was lowest (83 nm) due to a varying machining process using diamond wheels. Polishing of all surfaces significantly decreased S_a to 9–13 nm with no differences between materials. As polishing methods reach a limit defined by grain boundary relief and micro-chipping, these surface roughness S_a values of 9–13 nm represent a practical lower boundary for mechanical polishing of zirconia. Subsequent heat-treatment did not further affect the surface roughness, also considering that the detectable limit of the confocal device was reached, which necessitates further investigation with high resolution methods to elucidate the effect of processing methods on nano-structural changes.

The biaxial flexural strength σ correlated with yttria-content with a 1.5-to-1.8-fold increase when comparing 3YZ to 5YZ within the respective group. For all materials, polishing reduced the surface roughness and eliminated scratches and defects potentially serving as crack-initiation points and therefore increased biaxial flexural strength. It is commonly known that polishing eliminates surface defects which are the cause for the loss of a materials' original strength. Polishing, however, also induces mechanical stress on the surface with a controversially discussed effect: some studies reported that the monoclinic phase is consequently increased [7,36,37], while others described that it transforms to tetragonal phase [2,22,26] or is just removed and the bulk composition is exposed. As surfaces with a higher amount of monoclinic or also cubic phase do no longer benefit from crack healing effects due to transformation toughening, a heat-treatment to recover the tetragonal phase can be adapted to enhance aging resistance [24]. In the present study, the heat-treatment reduced strength for 3 mol% materials, while the strength of 4YZ and 5YZ remained similar. This was surprising, as grain boundaries re-appeared after heat-treatment on all surfaces, potentially serving as cracks initiation points as they represent a mechanical weak point. It is unknown whether heat-treatment also alters the crystal phase composition within the bulk material as XRD measurements are only possible to a depth of 70 μ m. Unfortunately, there are currently no methods to assess the bulk as cutting of the specimens and surface preparation would ultimately also affect the crystal phase ratio. This data thereby indicates that materials of distinct yttria concentration respond differently to heat treatment, implying that processing parameters cannot be universally applied to control biaxial flexural strength. Physical properties need to be understood in multi-parameter experiments to make informed and evidence-based decisions on zirconia-based materials for each specific application.

The scratch test induced chipping for 4YZ and 5YZ materials at a critical force F_c of around 88 N, owed to the mechanisms elucidated for biaxial flexural strength values. Surprisingly, polishing did not prevent the occurrence of chipping. As 3Y-TZP materials are optimized for transformation toughening they contain a high proportion of metastable tetragonal grains, which transform to monoclinic under stress, blunting cracks and preventing chipping [17]. 4YZ and 5YZ showed increased amounts of cubic phase, which does not transform under stress, hence the surface was mechanically more brittle, and lacked the energy-dissipating toughening mechanism of the tetragonal phase during scratch test.

Despite the observed trends regarding the effects of material composition and surface treatments, the study's limitations are largely influenced by the martensitic characteristics of zirconia. The yttria-content as well as surface processing influenced the crystal phase composition and the resulting surface structure ultimately affecting the materials' strength. Although the intention was to include materials for various zirconia applications in dentistry, as all of these are in direct contact with soft tissue, it was not possible to standardize the manufacturing process across all samples. These materials have distinct processing routes, which are dictated by their intended clinical application. Consequently, it remains unclear whether certain observations should be attributed to variations in processing parameters or to differences in composition of the materials used. When processing restorative materials, it is important to make dental technicians and dentists aware that certain processing steps, such as polishing, can significantly increase the mechanical strength of a restoration and in addition, improve fibroblast interaction [38]. On the other hand, polishing also eliminates the crystal grain topography and affects the crystal phase composition, which may be beneficial for bone cell - material interaction [23,39]. This study demonstrates how this surface structure can be retrieved by heat-treatment.

Nevertheless, the findings highlight that surface and mechanical properties of zirconia are determined by the yttria-content, surface processing and resulting crystallographic composition, presence of grain boundaries and grain sizes on the surface and surface roughness in general. By polishing the surface and heat-treatment, it was possible to eliminate macro-structures and create zirconia surfaces with distinct grain sizes in the mid nanometer-range. As zirconia in the oral cavity is in direct contact with hard and soft tissue, it is of high interest to further study whether cells (e.g. osteoblasts, fibroblasts, macrophages) are reacting to these nano-structured surfaces. This will allow to choose appropriate raw materials and manufacturing processes to target the respective cell response.

5. Conclusion

By characterizing surface and mechanical properties of five different zirconia materials used in dentistry it was shown how bulk material properties and subsequent manufacturing steps correlate with chemical, mechanical, and surface properties. The materials varied in their yttria-content and surface processing (as-sintered, polished, polished and heat-treated at 1250 °C). Polishing of zirconia eliminated extended grains on the surface, while the grain structures were retrieved by subsequent heat-treatment. Nevertheless, the findings underline the complexity of zirconia in dentistry as the materials' properties are affected by inter-dependent variables which are challenging to study, control, or standardize in an isolated way. Ultimately, the results of this study will feed into approaching refined, simplified, and cost-effective manufacturing methods for zirconia dental implants and restorative materials.

CRedit authorship contribution statement

Nadja Rohr: Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Murat**

Pinto: Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **Tiffany Moraz:** Writing – review & editing, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **Romy Marek:** Writing – review & editing, Supervision, Project administration, Methodology. **Anne Géraldine Guex:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Michael de Wild:** Writing – review & editing, Supervision, Resources, Methodology, Funding acquisition, Conceptualization.

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Declaration of competing interest

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ceramint.2026.01.447>.

Data availability

The data supporting the presented results can be viewed upon reasonable request to the corresponding author.

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