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Original Article

Effect of airborne-particle abrasion and polishing on novel translucent zirconias: Surface morphology, phase transformation and insights into bonding

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Abstract

Purpose: The purpose this study was to investigate the effect of Kern´s air-borne particle abrasion protocol (KAPA) and polishing on two translucent zirconias (4Y, 5Y-zirconias) compared to a traditional zirconia (3Y-zirconia).

Methods: Two different surface treatments were analysed by X-ray diffraction (XRD) and interferometry 1) KAPA (0.1 MPa, 50 μm alumina, 10-12 mm distance, 15 sec and 30 sec and cleaning in ultrasound using isopropyl alcohol 99%) and 2) Clinical-delivery polishing paste (Zircon Brite, Dental Ventures, USA). Shear-bond strength tests (SBS's) were performed with a highly polished and virtually flat surface in combination with a 10-MDP based cement and a surface modified by KAPA in combination with zinc phosphate cement. The SBS was expressed in terms of MPa.

Results: The mean values for monoclinic content were 13 wt%, 7 wt% and 2 wt% for 3Y-, 4Y- and 5Y-zirconias respectively, no differences were found between 15 and 30 seconds. Polishing did not result in phase transformation to monoclinic phase in any of the zirconias. The rhombohedral phase was identified in all types of zirconias regardless of surface treatment. Shear-bond strength tests showed 5 MPa for polished/10-MDP based cement and 3 MPa for KAPA/Zinc phosphate. Statistically significant differences were found between the two different surface treatments but not between the types of zirconias.

Conclusions: KAPA for 15 sec seems to be equal to 30 sec regarding morphology and phase transformation. Sole micro-retention appears not to be fully responsible for the bonding phenomena of 10-MDP and zirconia that underwent KAPA.

Keywords: *Air-borne particle abrasion, Translucent zirconias, 10-MDP, Bonding, Rhombohedral phase.*

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1. Introduction

Zirconia-based restorations are increasingly being recognized as an alternative to metal-ceramic restorations. Estimated five-year clinical survival rates of metal-ceramic and zirconia-based single crowns seem to be statistically equal [1] . Modification of the inner surface of single crowns is a common practice to increase the retention area and/or micromechanical interlocking. This is especially appealing for zirconia-based single crowns, given that loss of retention is one of the most common clinical complications of zirconia-based single crowns (4% for a 5-year period of clinical service) [1]. Thus, modification of the inner surface of the crowns is thought to be beneficial to increase the cementation area and/or micromechanical interlocking.

However, due to the nature of zirconia, the glass-ceramics' specific protocol of etching and silanization is not feasible. Various methods have been suggested over the course of the recent years. Glass infiltration, laser treatments, silica coatings and different chemical treatments have been investigated [2]. Nevertheless, a particular procedure proposed by Kern et al, to increase clinical predictability (10 years follow up) has demonstrated promising results. The procedure involves airborne particle abrasion (APA) with 50 μm alumina particles at pressures from 0.1 to 0.2 MPa with a cleaning step in ultrasound using isopropyl alcohol in combination with a 10-MDP-based-cement or primer [3].

New types of zirconias have recently entered the market. These new zirconias are usually characterized with improved translucency compared to traditional formulations [4]. This increased translucency seems to be related with increased quantities of yttria (Y_2O_3). The content of yttria is also related to an increased content of cubic-phase zirconia.

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Nonetheless, there might be a decrease in the fracture toughness and flexural strength with increased cubic content [5].

Bonding to these novel translucent zirconias may be plausible following the same APA protocol and the selection of a 10-MDP-based cement [6]. However, the effect of KAPA in phase content and surface roughness has been limited to 3Y and 5Y-zirconias [7,8]. Moreover, the effect on phase transformation of a polishing system intended for clinical use has been only reported for 5Y-zirconias [9,10].

The aim of this study was to characterize the surface of three types of commercially available zirconias (3Y,4Y and 5Y) that underwent KAPA including an extended treatment (30 sec). Additionally, to provide an insight into the effect of the surface morphology and type of cement on the SBS to 3Y, 4Y and 5Y-zirconias. The surface of polished zirconias with a polishing paste intended for clinical delivery (ZirconBrite, Buffalo, USA) was also characterized.

Our null hypothesis was formulated as follows

- The KAPA protocol will not induce any change in surface morphology and phase change among the different types of zirconias compared to the polished specimens.
- Additionally, no differences in SBS will be observed among the different types of zirconias with respect to surface treatment or cements.

2. Materials and Methods

The materials used in the present study are detailed in Table 1, and the experimental workflow is presented in Figure 1. A traditional zirconia containing 3 mol % yttria (BruxZir, Glidewell, Newport Beach, USA) served as control. The other two translucent zirconias contained 4 mol % yttria (DDcube X2 HS, Dental Direkt, Spenge, Germany) and 5 mol % yttria (CopraSmile, Whitepeaks, Wesel, Germany). Each type of zirconia was sintered according to the manufacturers' instructions. Zirconia plates (10 mm x10 mm x 1 mm or 1.5 mm) were used. Prior to the XRD and interferometry measurements the plates were sequentially polished with abrasive paper: #500, #1000, #2000, #2400 and #4000 (Struers ApS, Bromma, Sweden) until optical finish on both surfaces (to ensure a similar starting surface), with the last polishing step performed using a soft bristle brush (Buffalo Dental, Syosset, USA) with Zircon-Brite polishing paste (Dental Ventures, Corona, USA). One surface underwent KAPA at a pressure of 0.1 MPa using an APA device (Basic professional, Renfert, Hilzingen, Germany). The final cleaning step was performed in ultrasound using isopropyl alcohol 99% for 15 minutes.

2.1. Surface Morphology (Interferometry)

Prior to the XRD and SBS experiments, the surface morphology of the different types of zirconia was assessed using interferometry.

Three specimens per type of zirconia (n=3) at three different points per specimen were analyzed (9 measurements per type of zirconia). The information was acquired using a white-light interferometer (SmartWLI extended, Gbs, Germany). The parameters of interest were three: (a) Sa (µm) i.e. average roughness, (b) Sdr (%) i.e. additional surface area contributed by the roughness and (c) Sds (1/µm2) i.e. density of summits.

The specimens were scanned using a 50X Mirau objective with a height resolution of 0.1 nm. Both surfaces, KAPA and polished, were scanned. An anti-vibration device was activated (Nanoseries, Accurion, Germany). The measured area was 350×220 µm. The acquisition of the data was performed by the SmartVIS3D software version 2.1 (Gbs, Germany) and processed using the MountainMaps software version 7.4 (Digital Surf, France). The data was processed using a high-pass Gaussian filter of size 50×50 µm as suggested by Wennerberg and Albrektsson [11]. The analysis of the surface profile (Fig. 9) was performed using the software ImageJ version 2.0-rc-43 (Open source image processing software, Creative commons license).

Table 1. Materials used in the present study .

Material	Trade Name	Manufacturer
3Y-zirconia	BruxZir 2.0 (lot B1226355)	Glidewell, USA
4Y-zirconia	DDcubeX^2 HS (lot 6161719002)	Dental Direkt, Germany
5Y-zirconia	CopraSmile (lot IS2189A2)	Whitepeaks, Germany
10-MDP-based cement	Panavia F 2.0, Paste A lot 7E0167 Paste B lot 270072	Kuraray, Japan
Zinc phosphate cement	Liquid, Normal setting lot 1101609 Powder, Normal setting lot 91605022	Harvard Dental International, Germany

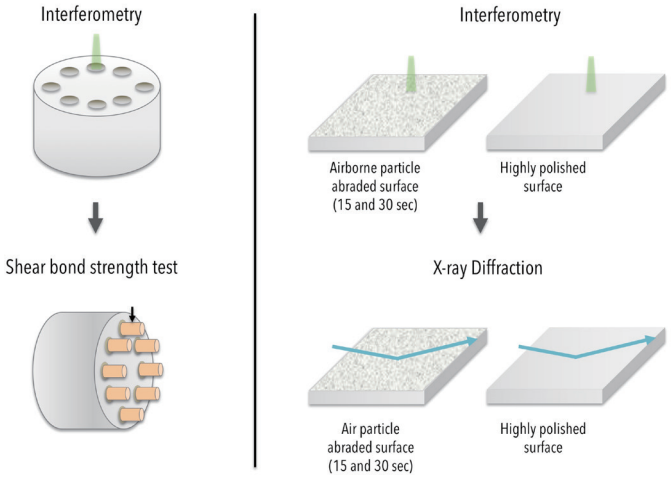

Fig. 1. Illustration of the experimental setting of the study.

2.2. XRD

Three plates per type of zirconia (n=3) and surface treatment underwent XRD. Information from each plate was acquired using a SmartLab diffractometer (Rigaku, Tokyo, Japan). The diffractometer used Cu Kα radiation as the x-ray source at 45 kV of voltage with a current of 200 mA. The step size was 0.02° with a scan speed of 10°/min. The phase contribution was obtained using the Rietveld analysis [12] implemented in the TOPAS 5 program [13].

All models of the crystal structures used in the analysis were taken from the ICSD data base (https://icsd.fiz-karlsruhe.de/search/basic.xhtml) and their details can be accessed elsewhere [14–17] for the cubic (c), tetragonal (t), rhombohedral (r) and monoclinic (m) phases, respectively. During the refinement the atomic positions and thermal parameters were fixed whereas the phase contribution and the lattice parameters for all phases were refined. The intensities of the cubic phase in the patterns for the specimen that underwent KAPA required a texture correction that was applied using spherical harmonics implemented in TOPAS. Additionally, the monoclinic volume fraction (Vm) was estimated by the Garvie and Nicholson method [18] modified by Toraya [19]:

$$Vm = 1.311 \times Xm / (1 + 0.311 \times Xm)$$

$$Xm = [Im(-111) + Im(111)] / [Im(-111) + Im(111) + It(101)]$$

It and Im represent integrated intensities of the tetragonal and monoclinic phases at different peaks. The software PDXL (Rigaku, Japan) was used to estimate the integrated intensity of each. The monoclinic phase volume fraction was presented as the percentage of the tetragonal phase.

2.3. Shear-bond strength test

Thirty zirconia cylinders (3.0 mm φ x 2.0 mm height) were prepared for the shear-bond strength test(SBS). Ten specimens were used for each

type of zirconia (n=10) and subsequently embedded in resin (EpoFix batch 8348-01, Struers, Ballerup, Denmark) according to manufacturer's instructions. The traditional zirconia (3Y-zirconia) served as control material.

The embedded zirconia cylinders were polished using SiC papers (Struers, Ballerup, Denmark) under constant water rinsing. The following grit size sequence was used #500, #1000, #2000, #2400 and #4000. The last step was carried out using a polishing cloth in combination with a lubricant (DP-Lubricant Blue, batch 5335, Struers, Ballerup, Denmark) and a polishing suspension (OP-S Suspension, batch 4218-8333, Struers, Ballerup, Denmark). After polishing, the embedded zirconia cylinders were rubbed for 3-4 sec with isopropyl alcohol 99% and left to dry at room temperature. Subsequently, the zirconia cylinders were analysed using the same instrumentation and followed the same protocol as for the zirconia plates.

Thirty polymerized composite resin cylinders (2.4 mm φ x 1.5 mm height) (Spectrum TPH3 lot 0986, Dentsply Sirona, Charlotte, USA) were cemented onto the surface of the polished cylinders. A 10-MDP-based cement (Panavia F 2.0 cement, paste A lot 7E0167, paste B lot 270072, Kuraray, Kurashiki, Japan) was used. A defined cementation area was ensured by removal of cement excess. The cement and the composite resin cylinders were light-cured using a LED lamp (Lumion, Planmeca, Helsinki, Finland) with an approximate irradiance of 850 mW/cm2. The specimens were left undisturbed at room temperature for 20 min before being immersed in deionized water at 37°C for 24 hours.

The shear bond strength test was performed based to the ISO 29022:2013. Each epoxy resin cylinder containing 10 specimens of the same type of zirconia was mounted and secured in a universal testing machine (LRX 9772, Lloyd Instruments, Bognor Regis, UK). A notched-edge cross head at a rate of 1.0 mm/min was used. The shear bond strength was calculated by dividing the force at debonding (N) by the bonding area (4.52 mm^2). After the first SBS test, the surface of the zirconia cylinders was cleaned from remaining cement and then immersed in isopropyl alcohol 99% for 15 minutes in ultrasound.

The surface was visually inspected for any residual cement. Later,the surface underwent KAPA. The cylinders were again cleaned with ultrasound using isopropyl alcohol 99% for 15 minutes. Thirty zinc phosphate cylinders (2.4 mm ϕ x 1.5 mm height) were produced using a PMMA mould with the same dimensions. The cylinders were then cemented on the KAPA treated surfaces, 10 per type of zirconia, followed by careful removal of cement excess using an explorer. The specimens were left undisturbed for 20 min before being immersed in deionized water at 37°C for 24 hours. The second SBS was carried following the same ISO-based protocol as the initial SBS.

2.4. Statistics

A Shapiro-Wilk test was performed in order to assess the normality of the data, both from interferometry and SBS. As the data was found to be normally distributed a one-way ANOVA test was performed. The ANOVA test was accompanied by a Tukey HSD and a paired t-test (same type of zirconia but different APA time) for the interferometry. The SBS results were compared with the ANOVA test and complemented by a Dunnett test 3Y-zirconia as control.

3. Results

3.1. Surface morphology

The results summarizing the surface morphology are shown in Table 2. The KAPA group presented a mean surface roughness (Sa) value of 0.40 (range 0.38-0.42 μm). Surface developed ratios (Sdr) resulted in a mean value of 125 (range 116-134%). The APA treated 5Y-Zirconias had a mean value of 134 (range 133-134%). Their mean value of the density of summits (Sds) was 1.62 (range 1.59- 1.64 /$μm^2$). Statistically significant differences were found within both groups, 15 sec and 30 sec (Table 2). No statistically significant differences were observed in surface roughness (Sa) between 15 sec and 30 sec surface treatments, in any of the types of zirconias (Table 3). However, the 4Y-zirconia showed statistically significant higher mean values for, Sdr and Sds. The polished specimens presented no statistically significant differences in Sa. Statistically significant differences were found for Sdr in the polished specimens. A visualization of each surface treatment is presented in Figure 2.

3.2. XRD

The XRD results are presented in Table 4. The analysis of the diffraction patterns showed three phases (c, t and r) in all polished samples. Figures 3 and 4 show a representative fit to the polished 4Y-zirconia specimen as well as contributions from each phase for three selected Bragg peaks. As can be seen, the main contribution originates from the c and t phases. However, there is a small hump around 2θ = 29.5° which can be assigned to a small amount of the r phase. The intensity of the peak around 2θ = 29.5° increases significantly for the KAPA group as seen in Figures 5 and 6. It is also seen that for the KAPA specimens a new peak around 2θ = 28° appears. This peak can be assigned to the monoclinic phase. The monoclinic phase content (wt% and Vm) after KAPA was inversely related to the amount of yttria in the zirconias (3Y-zirconia>4Y-zirconia>5Y-zirconia). No monoclinic phase was found in the polished samples by either Rietveld refinement or the Garvie and Nicholson method modified by Toraya. The detailed contribution of each phase is listed in Table 4.

3.3. Shear-bond strength test

Table 5 illustrates the findings of the SBS tests. No statistical differences were found in the "polished surface/10-MDP based cement". The mentioned group had an approximate SBS of 5 MPa. The group "KAPA/ Zinc phosphate cement" presented an approximate SBS of 3 MPa. Statistically significant differences were found between 3Y-zirconia and 4Y-zirconia (Fig. 7). Significant differences between "polished surface/10-MDP-based cement" and "KAPA/ Zinc phosphate cement" (Fig. 7 and Table 6).

4. Discussion

Bonding to traditional zirconia (3Y-zirconia) that underwent APA via a 10-MDP-based cement was suggested as early as 1998 by Kern [20]. Ten-year clinical findings for 10-MDP-bonded traditional zirconia 3Y-zirconia restorations were published in 2017. The clinical study used a similar APA protocol as proposed by Kern in 1998 (0.1-0.25 MPa, 50 μm alumina, ultrasound cleansing in isopropyl alcohol 99%), but no description on the distance or time during APA was provided [21] . To the best of our knowledge, the present study investigated for the first time the effect of KAPA on different type of zirconias with an extended treatment period (30 sec).

The present study found similar values of the effect of KAPA on the monoclinic phase content for 5Y-zirconias and 3Y-zirconias [7,8]. However, those studies used at least double the pressure as compared to the pressure used in the present study (0.2- 0.25 MPa) and did not report an extended APA period. Nevertheless, in the present study and the mentioned studies, the monoclinic wt% in 5Y-zirconias was about 2% and the monoclinic Vm about 10% for 3Y-zirconia. Other studies hat have used larger sized alumina particle, i.e. 110 μm for 3Y-zirconia, however similar values to the ones found in the present study was reported. About 14% wt% and 15% Vm monoclinic phase [22,23].

Table 2. Surface Morphology results.

I. APA 15 seconds, 0.1 MPa (1 Bar), 10-15 mm distance			
Material	Sa, µm (SD)	Sdr, % (SD)	Sds, 1/µm^2 (SD)
3Y-zirconia	0.39 (0.01) [a]	122.07 (3.17) [a]	1.61 (0.03) [a]
4Y-zirconia	0.38 (0.02) [a]	121.20 (3.04) [a]	1.64 (0.02) [b]
5Y-zirconia	0.41 (0.15) [b]	133.02 (2.15) [b]	1.62 (0.01) [b]
II. APA 30 seconds, 0.1 MPa (1 Bar), 10-15 mm distance			
Material	Sa, µm (SD)	Sdr, % (SD)	Sds, 1/µm^2 (SD)
3Y-zirconia	0.39 (0.01) [a]	120.52 (3.24) [a]	1.63 (0.01) [a]
4Y-zirconia	0.38 (0.01) [b]	116.33 (7.21) [a]	1.59 (0.04) [b]
5Y-zirconia	0.42 (0.01) [c]	134.21 (6.19) [b]	1.61 (0.02) [b]
III. Step-wise polished, ZirconBrite paste, last step: soft bristle brush			
Material	Sa, µm (SD)	Sdr, % (SD)	Sds, 1/µm^2 (SD)
3Y-zirconia	0.009 (0.001) [a]	0.02 (0.01) [a]	0.64 (0.11) [a]
4Y-zirconia	0.011 (0.004) [a]	0.04 (0.02) [b]	0.68 (0.06) [a]
5Y-zirconia	0.009 (0.002) [a]	0.02 (0.01) [a]	0.71 (0.08) [a]

SD: standard deviation
Means that don't share the same superscript are statistically different according to the Tukey´s HSD test.

Table 3. Paired t-test, 15 seconds APA – 30 seconds APA.

	Variable		
Pair	Sa	Sdr	Sds
3Y-zirconia 15 sec – 3Y-zirconia 30 sec	p= 0.802	p= 0.322	p= 0.105
4Y-zirconia 15 sec – 4Y-zirconia 30 sec	p= 0.553	p= 0.045*	p= 0.003*
5Y-zirconia 15 sec – 5Y-zirconia 30 sec	p= 0.439	p= 0.541	p= 0.139

*: Statistically significant difference (p<0.05)

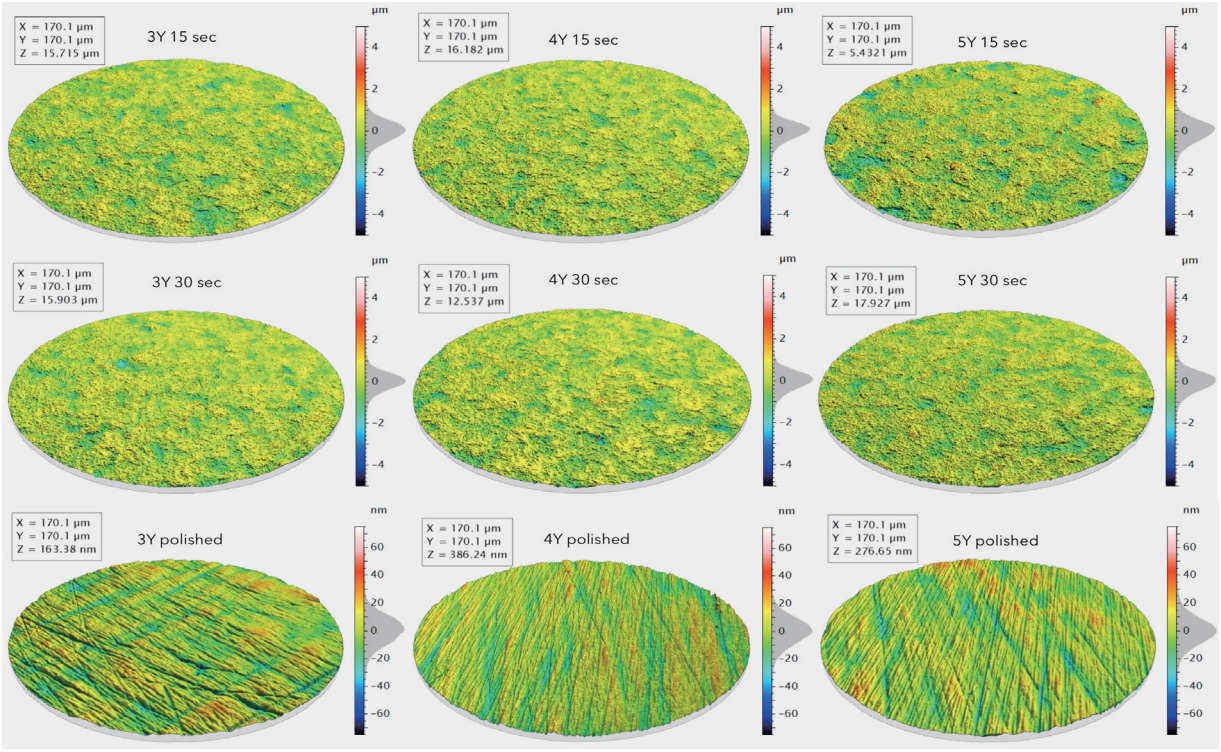

Fig. 2. Illustration of the surface morphology findings. A significant difference was observed between 15 sec and 30 sec among all types of zirconias. Polishing marks on the nanometer scale can be observed on the polished specimens.

The identification and reporting of the rhombohedral phase is not common in dental research. The rhombohedral phase was reported as early as 1977 by Scott [16] and recently reported in dental research literature in a number of articles for 5Y and 3Y-zirconias [7,24,25]. Other articles related to ceramic engineering have reported the identification of this phase in different yttria-stabilized zirconias (2.5Y, 3Y, 4Y, 5Y, 7Y and 10Y-zirconias) [26–29]. This phase seems to be characterized by the broadening of certain peaks compared to as-sintered or polished specimens (Fig. 5 and 6).

Some authors regard it as a distorted tetragonal phase [24,25,29], others as a distorted cubic phase [7,27], yet some as originating from both distorted tetragonal and cubic phases [7,26]. However, it has been suggested that the rhombohedral phase may not be a phase per se, but a reorientation of the domains within the crystal unit, specifically, ferroelastic switching with the possibility to reverse to tetragonal or cubic after annealing [30]. Nevertheless, the volume of the rhombohedral phase unit cell seems to be 2.5 % larger compared to the cubic phase unit cell [31] and 1-2% larger than the tetragonal unit cell [29]. Likewise, its crystal unit may resemble a distorted fluorite-like structure along the 111 plane [31] (Fig. 8). The high rhombohedral phase content (>50% wt%) reported for the KAPA group in the present study may be due to different factors. The pressure used (0.1MPa) could be enough to only partially remove some

Table 4. XRD results.

	wt% (SD)				Vm (SD)
Material -- treatment	Cubic	Rhombohedral	Tetragonal	Monoclinic	Monoclinic
3Y-zirconia -- 15 sec	19% (1)	55% (1)	13% (1)	14% (1)	10% (4)
3Y-zirconia --30 sec	18% (1)	58% (1)	13% (1)	13% (1)	7% (0.2)
3Y-zirconia -- Polished	23% (1)	16% (1)	62% (1)	0%	0%
4Y-zirconia -- 15 sec	24% (1)	60% (1)	9% (1)	7% (1)	3% (0.1)
4Y-zirconia -- 30 sec	24% (1)	60% (1)	9% (1)	7% (1)	2% (0.3)
4Y-zirconia -- Polished	37% (1)	17% (1)	51% (1)	0%	0%
5Y-zirconia -- 15 sec	26% (1)	68% (1)	9% (1)	2% (1)	1% (0.5)
5Y-zirconia -- 30 sec	27% (1)	67% (1)	9% (1)	2% (1)	1% (0.3)
5Y-zirconia -- Polished	49% (1)	13% (1)	38% (1)	0%	0%

Vm: Volume fraction determined by Garive and Nicholson (modified by Toraya)
wt%: Weight percentage determined by Rietveld Refinement
SD: Standard deviation

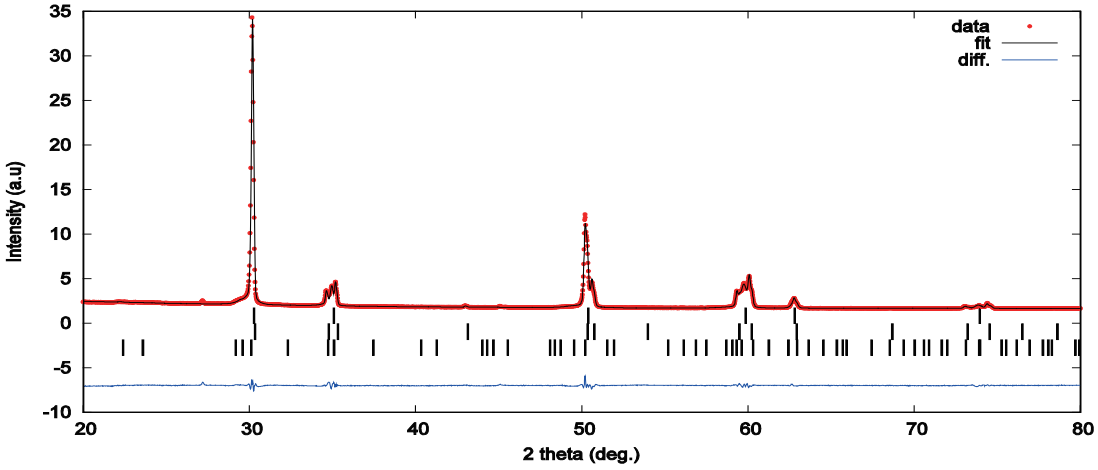

Fig. 3. Diffractogram of a polished 4Y-zirconia. Red dots represent the data points, the black line is a fit and a blue line shows a difference between the model and the data.

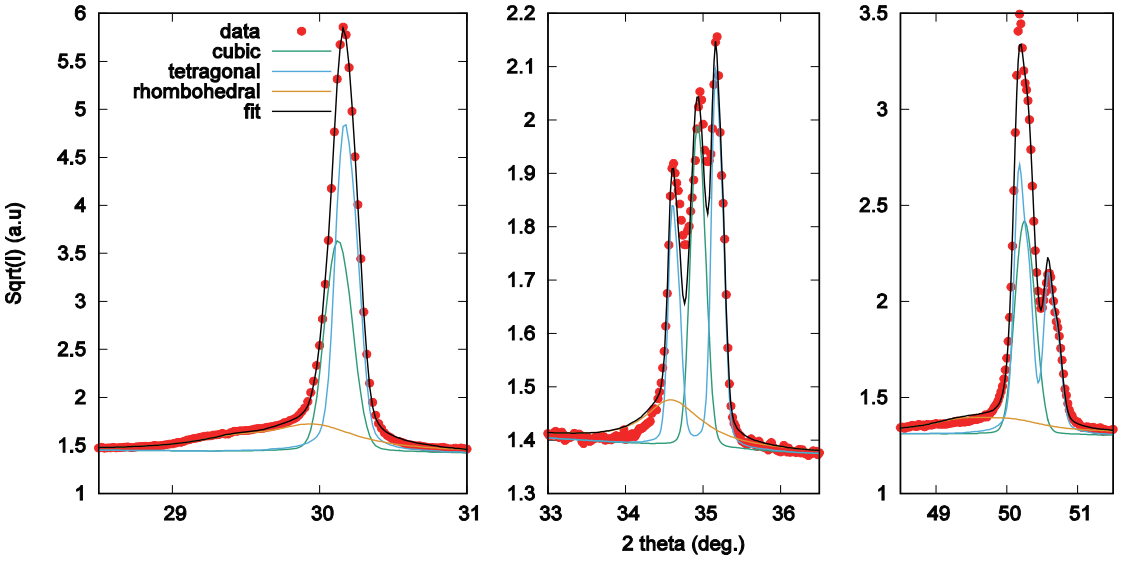

Fig. 4 . Selected diffractogram peaks of a polished 4Y-zirconia. Cubic (green line), tetragonal (blue line) and rhombohedral (orange line) phase contribution for the selected Bragg peaks in the diffraction pattern collected for the sample 4Y-polished.

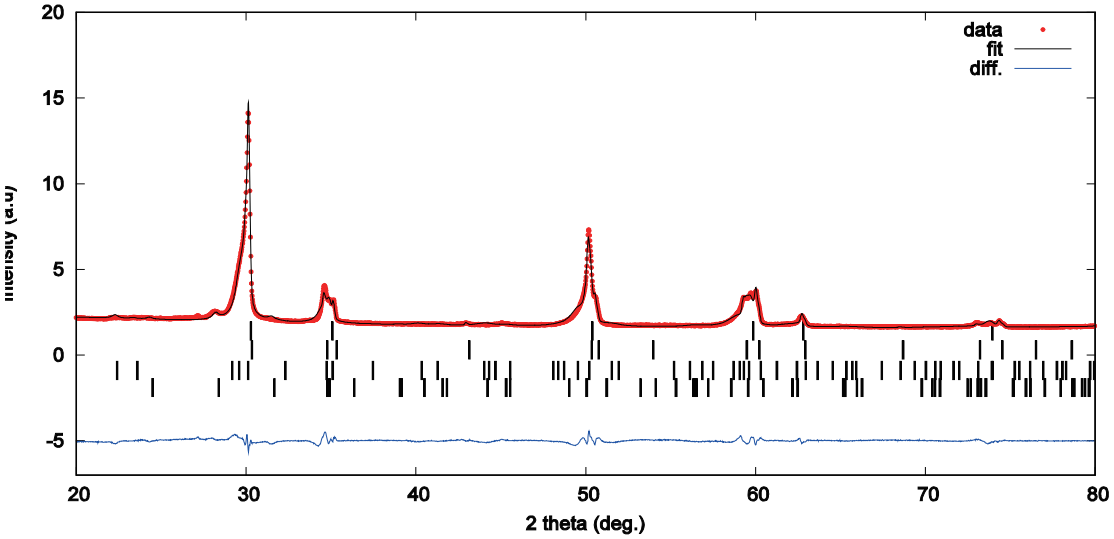

Fig. 5 . Diffractogram of an air-borne particle abraded 4Y-zirconia according to the KAPA protocol. Red dots represent the data points, the black line is a fit and a blue line shows a difference between the model and the data.

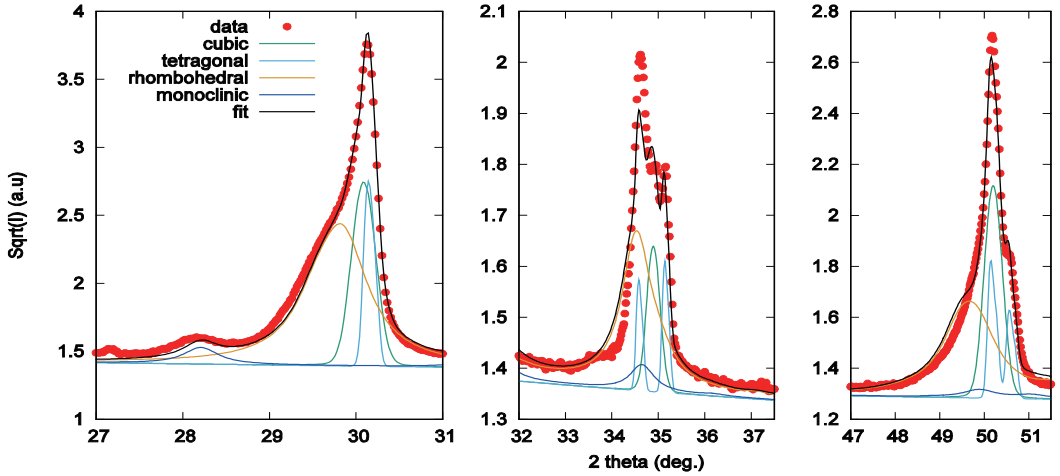

Fig. 6 . Selected diffractogram peaks of an air-borne particle abraded 4Y-zirconia according to the KAPA protocol Cubic (green line), tetragonal (light blue line) and rhombohedral (orange line) and monoclinic (dark blue line) phase contribution for the selected Bragg peaks in the diffraction pattern.

Table 5. Shear bond strength (SBS) and Surface Morphology findings.

	SBS 24 h		Surface Morphology		
Material//Cement	Mean, MPa (SD)	95% CI	Sa, µm (SD)	Sdr, % (SD)	Sds, 1/µm^2 (SD)
3Y-zirconia//Zinc Phosphate (Control in Dunnett test)	5.1(1.7)	3.9 - 6.4	0.03 (0.003)	5.84 (1.87)	0.50(0.02)
4Y-zirconia//10MDP based cement	4.8 I(1.2)	4.0 - 5.7	0.03 (0.005)	6.00 (1.30)	0.40(0.02)
5Y-zirconia//10MDP based cement	6.0 I(1.2)	5.1 - 6.8	0.06 (0.013)	13.90 (4.90)	0.60(0.09)
5Y-zirconia//10MDP basedcement	3.6(0.4)	3.2 - 3.9	0.41 (0.005)	134.15 (2.01)	1.66(0.01)
4Y-zirconia//Zinc Phosphate	2.1II(0.9)	1.3 - 2.9	0.40 (0.008)	126.55 (2.78)	1.64(0.01)
5Y-zirconia// Zinc Phosphate	2.9I(0.7)	2.3 - 3.5	0.39 (0.004)	119.21(2.42)	1.63(0.01)

CI: Confidence Interval, SD: standard deviation, I: p>0.05 Dunnett test, II: p<0.05 Dunnett test

superficial zirconia layers and generate some monoclinic phase, but also, low enough to provide the necessary energy to deform and not remove some tetragonal and cubic crystal units. Furthermore, it could also be related to the texture correction implemented in the Rietveld refinement software (TOPAS 5). However, the monoclinic wt% was comparable to those found in the literature for 5Y and 3Y-zirconias. As mentioned earlier and according to the best of our knowledge, this is the first time that phase quantification of different zirconias is performed after KAPA treatment at a pressure of 0.1MPa or 10 gf/mm^2. That pressure was the lowest pressure that the APA device used

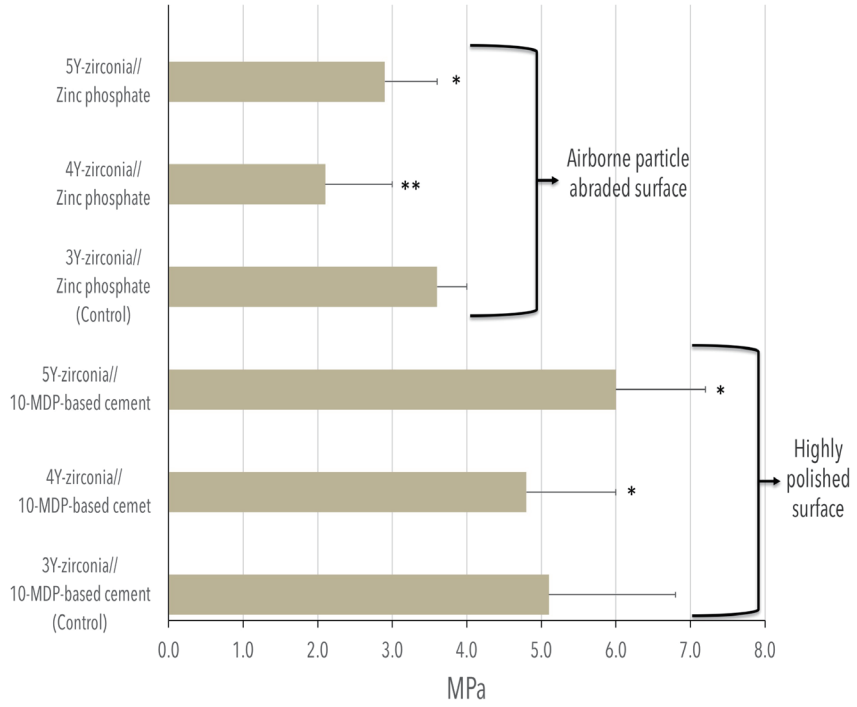

Fig.7. Chart of the shear bond strengths, *: p>0.05 Dunnett test, **: p<0.05 Dunnett test

Table 6. Paired t-tests, 10-MDP based cement (polished surface) -- Zinc Phosphate (APA´d surface).

Pair	SBS (MPa)
3Y-zirconia//10MDP based cement -- 3Y-zirconia //Zinc Phosphate	p= 0.012*
4Y-zirconia//10MDP based cement -- 4Y-zirconia//Zinc Phosphate	p= 0.000*
5Y-zirconia//10MDP based cement -- 5Y-zirconia//Zinc Phosphate	p= 0.000*

*: Statistically significant difference (p<0.05)

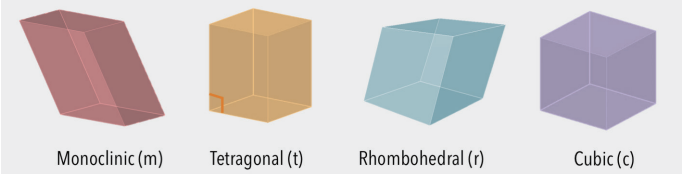

Fig. 8. Illustration of the different phases found in yttria-stabilized zirconia formulations

in the present study (Basic professional, Renfert, Germany) could produce. The polished specimens showed no monoclinic content as has been reported for 3Y and 4Y-zirconias [9,10,32]. However, in the present study it was reported that some rhombohedral phase was formed after polishing (13- 17% wt%) and could be related to the inherent mechanical challenges of polishing on the superficial layers leading the distortion of some tetragonal and cubic crystal units.

The surface morphology was consistent among the different types of zirconias, about 0.4 µm for all types of zirconias for both 15 and 30 sec of APA treatment. Values reported in the literature for 5Y and 3Y-zirconias vary from 0.2 to 0.5 µm at pressures of 0.2 to 0.28 MPa with 50 µm alumina for 15 sec [7,33]. However, the mentioned studies used different methods for the acquisition of the morphological information. Namely, a confocal laser scanning profilometer [7] and a contact stylus profilometer [33]. In the present study three types of zirconias were investigated using white light interferometry, which has a higher horizontal and vertical resolutions than thetwo above mentioned methods [11]. However, all three methods seem to be acceptable for blasted and polished surfaces [11].

One particular variable, surface developed ratio (Sdr), was of special interest in this study. The Sdr indicates how much extra surface area that is produced by the surface roughness. Sdr values ranged between 116-134 %, meaning that for every 1 mm2 another ~1.2 mm^2 are created through KAPA. The SBS on a virtually flat surface (Sa: ~0.004 µm, Sdr: ~8%, Sds: ~0.50/µm2) were 5 MPa,probably due to the chemical interaction between the 10-MDP-based cement and the zirconias. Shear-bond strength values, to APA treated surfaces, of about 20 MPa can be found in another study where the same type of zirconias, 10-MDP-based cement, composite cylinder and experimental conditions [6]. Nevertheless, the mechanical properties of resin-based cements differ compared to zinc phosphate cement [34].Specifically, the lower flexural strength and compressive strength of zinc phosphate compared to the resin 10-MDP-based cement [34] may have influenced the debonding of the cylinders and therefore the SBS values, that difference may have hindered the comparison between groups. However, the higher elastic modulus of zinc phosphate compared to resin cements [34] might have provided additional resistance to deformation at the moment of debonding.

According to the Inorganic Crystal Structure Database (ICSD) (https://icsd.fiz-karlsruhe.de/search/basic.xhtml) the cell volume of largest crystal unit zirconia (monoclinic) is 140 Å3. A conservative calculation could illustrate that every 140 Å3 there is at least one Zirconium4+ ion for bonding. Hence, it could be speculated that APA does not only produce an increased surface area and microretention but also provides, three-dimensionally, more potential bonding sites (See Fig. 9).

5. Conclusion

The KAPA protocol resulted in an increased surface roughness and a phase transformation within the zirconias. A prolonged KAPA treatment period from 15 to 30 sec gave no additional effect on the surface

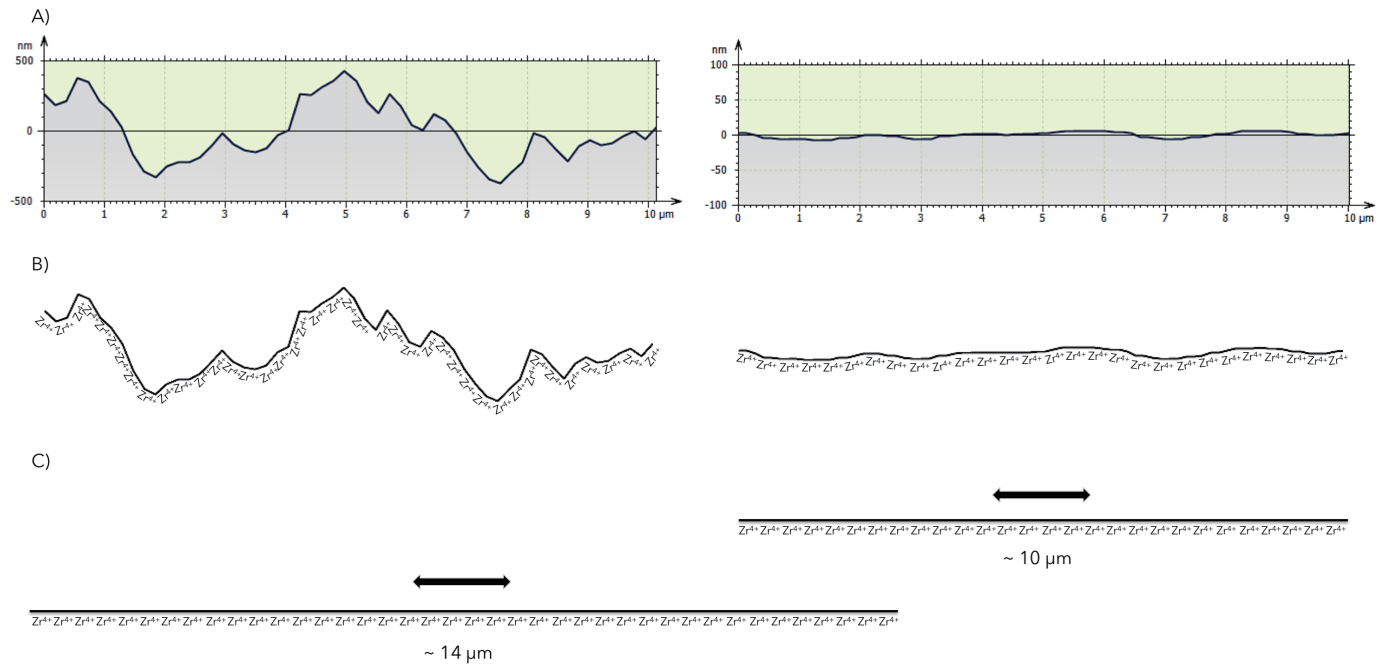

Fig. 9. Insight into the effect of air-particle borne abrasion on the surface of yttria stabilized zirconias .The air-borne particle abrasion increases the surface area and possibly the available bonding.

morphology or phase transformation. The null hypothesis was rejected regarding the effect on the KAPA treatment on the phase transformation and partly the surface morphology. The null hypothesis regarding the shear-bond strength was partially rejected, since significant differences were found between surface treatment and cement but not between the types of zirconia.

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Conflict of interest

The authors report no conflict of interest

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