

Comparison of Polishing Effects of Different Polishing Systems on Highly-Translucent Monolithic Y-TZP Ceramics

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Abstract: To evaluate the polishing effect of different polishing systems on different brands of highly-Translucent Monolithic Y-TZP Ceramics. Four brands highly-translucent zirconia ceramic were selected to make specimens with the size of 15mm x 10mm x 5mm, and the surface was roughened and ground by simulating the clinical procedure, followed by polishing with one of the four polishing systems respectively. Then the surface roughness of each specimen was determined using non-contact laser scanning profilometer. Finally, the surface microstructure was observed using SEM and the crystal phase transformations in the surface were assessed using XRD and XPS. EVE polishing system was more effective than the other systems, and the diamond abrasive particle size in its fine polishing bur is the smallest and evenly distributed. The XRD results showed that surface roughening and the following polishing processes did not lead to obvious phase transformation of surface tetragonal zirconia. XPS spectrogram shows that, the following processes have a certain effect on the structure performance of zirconia.

1. Introduction

Zirconia is a commonly used material for fixing dental defects and missing teeth in clinical practice. Its good mechanical properties, aesthetics and excellent biocompatibility make it popular among clinicians and patients, and it has become the mainstream fixed crown bridge repair material. At present, the main materials used for making all-ceramic crowns are aluminum oxide glass penetrant ceramics, hot die casting glass ceramics and yttria-stabilized tetragonal zirconia polycrystals (Y-TZP). Among them, the mechanical properties of Y-TZP porcelain stabilized by 3%mol yttrium are significantly better than other ceramic materials, coupled with its excellent biocompatibility, Y-TZP porcelain has been widely used in the production of post-core, orthodontic bracket, artificial implant materials and implant abutters in the dental pulp cavity through CAD/CAM technology [1-4]. To prolong the service life of zirconia all-ceramic restorations, a monolithic zirconia crown-bridge restoration with porcelain decoration removed was designed.

With the development of the production process, the transparency, coloring mode and coloring stability of the monolithic zirconia restoration were improved. Its visual aesthetics has been significantly improved, so it has gradually been widely used in clinical practice in recent years. However, when the monolithic zirconia prosthesis is tried in clinical practice, it needs to be adjusted according to the needs of treatment. After grinding, the surface roughness of the monolithic zirconia prosthesis causes the mechanical strength to decrease, the wear on the antagonist teeth is increased, and the rough surface is more likely to be colored, adhere to bacteria and increases the risk of low temperature degradation (LTD) of zirconia materials. Therefore, the surface polishing treatment after clinical adjustment of zirconia is particularly important. Polished zirconia restoration can reduce the accumulation of plaque, improve patient's comfort, remove defects that may lead to fracture, and smooth the surface contacted with opposite enamel[5]. Hjerpe J et al. found through the three-point bending experiment that there was a significant negative correlation between the surface roughness of zirconia specimen and its strength [6], and polishing could reduce the surface defects of zirconia and improve the strength of the restoration. In addition, long-term exposure of restorations with rough surfaces to oral moisture may also promote low temperature degradation (LTD) of zirconia materials [7,8]. The surface grains in contact with water molecules slowly change from tetragonal phase to monoclinal phase, resulting in reduced bending strength. It affects the long-term prognosis [9]. Low temperature aging of zirconia occurs over time in the temperature range of 65 °C to 500 °C, immersed in water and other solvents [10] Although this mechanism is very slow at oral temperature, zirconia restorations are exposed to other factors such as constant humidity, heat changes, pH changes, chewing and repeated high occlusal load due to dysfunction. These factors can accelerate the aging process and reduce the bending properties of the material [11,12]. It has been reported [13] that a well-polished zirconia surface not only has low abrasion behavior on the antagonistic teeth, but also has good biocompatibility, which can reduce the adhesion of bacteria and plaque. Some research results have shown that the phase transformation of zirconia surface is directly related to the damage caused by surface treatment, and many stimulus factors can lead to the formation of small cracks, such as thermal change, humidity, particle wear and grinding in the air, etc., while the area with surface defects and defects is a potential place for crack initiation and expansion to the stress concentration area [14]. Under different ambient temperature conditions, there are three crystal phases of zirconia. It has been reported that clinical grinding of the contour and bite of zirconia restorations provides external energy through local heat and friction generation in zirconia, resulting in local M-phase transformation [15,16].

The commonly used surface smoothing treatment methods in clinical practice are glazing and polishing, but the glaze layer after glazing is easy to wear during chewing, and studies have shown [17,18] that the surface roughness after polishing Y-TZP porcelain is significantly less than that after glazing. In addition, Y-TZP porcelain has high mechanical strength, so the wear surface of the restoration can maintain a small roughness for a long time. In the process of grinding and polishing, the maximum surface temperature of zirconia can reach 2726.85°C[19], and the temperature reached in conventional grinding and polishing is slightly higher than 726.85°C. Due to the low thermal conductivity of zirconia, surface phase transformation may occur under the action of local high temperature, and the surface morphology of zirconia will also change.

2. Methods and materials

Adopting four mainstream brands of zirconia blocks in China's domestic market: Sagemax (NexxZr +, Sagemax, USA); Eltron (UPCERA ST, Upcera, China); Cercon ht (Dentsply, USA); Audental DUT (Audental, China)(Tab.1). The 4 manufacturers of zirconium blocks had been reduce into 15 millimeter x 10millimeter x 5 millimeter by way of the usage of an

electronically controlled accuracy diamond saw noticed (Well, Walter Ebner, Switzerland) with a width of 0.17 millimeters in addition a unpolished of 30 μ m beneath steady water cooling, every manufacturer of zirconium blocks used to be made of 60 blocks. Then the specimens had been polished with 2400 μ m corundum paper (Streuers, Willich, German) below prerequisites till a flat-faced used to be acquired. The samples were sintered at 1600 $^{\circ}$ C in a Zirconia crystal furnace (SINTR, Israel) for 16 hours.

Table 1: Four mainstream brands of zirconia blocks in China's domestic market.

Brand	Manufacturer	Chemical composition	Mechanical and physical properties
Upcera	UPCERA ST,Upcera,China	ZrO ₂ >98%,Y ₂ O ₃ <0.5%,HfO ₂ <0.1%,Fe ₂ O ₃ <0.3%	flexural strength:1200 MPa fracture toughness: 5 MPa/m ²
Cercon	Cercon ht,Dentsply,America	ZrO ₂ >91.9%,Y ₂ O ₃ <5%,HfO ₂ <2%,Al ₂ O ₃ +SiO ₂ <1%	flexural strength> 1200 MPa fracture toughness > 5 MPa/m ²
Sagemax	Sagemax Bioceramics, Sagemax, USA	ZrO ₂ +HfO ₂ +Y ₂ O ₃ \geq 99%; HfO ₂ \geq 1~5%;Y ₂ O ₃ >7.0~9.0%	flexural strength: 1000 MPa fracture toughness \geq :5 MPa/m ²
Audental	Audental DUT Audental, China	ZrO ₂ +HfO ₂ +Y ₂ O ₃ \geq 99%, Al ₂ O ₃ \leq 0.05%;Y ₂ O ₃ >4.5~6.0 %	flexural strength \geq 800MPa fracture toughness \geq : 6 MPa/m ²

Each brand zirconia block was randomly divided into 6 groups, each group produced 10 zirconia specimens, and 4 brands produced 200 zirconia specimens. The clinical up-grinding procedure was simulated. The first group was the blank control group, and the other 4 groups were first polished with 40 μ m emery needle (TR-13EF, MANI, Japan) and connected to high-speed mobile phone (Bein-Air, Switzerland) with light pressure water injection for surface roughening treatment (GB group). Then the final polishing procedure is carried out using different polishing systems, KOMET Group: Gumei Zirconia polishing set; EVE Group: EVE zirconia polishing set; Tob Group: TOBOOM Zirconia polishing set; SF Set: Matsufeng Zirconia polishing set, connected to electric sander (MIOX, NSK, Japan), set speed of 1,000rap/min, under the condition of no water injection, each step of the polishing procedure is performed for 2 minutes,. After polishing, the specimen was put into the ultrasonic cleaning machine, and the sample was ultrasonic cleaned in distilled water for 10 min. Whole programs have been carried out via the identical dentist with extra than 10 years' experience.

2.1 Surface roughness and Surface characterization analysis

A 3D topography scanner (PS-50, Nanovea, USA) was used to measure the surface roughness of each group of zirconia specimens and perform 3D digital topography reconstruction of the surface of the scanned area. The surface morphology of the specimen was characterized by scanning electron microscope (SEM), and the polishing heads of four kinds of polishing systems were observed under electron microscope. The valence states and approximate contents of chemical elements before and after polishing were detected by X-ray photoelectron spectroscopy (XPS), and the vacuum degree in the XPS analysis chamber was required to be $<5 \times 10^{-8}$ Pa. X-ray diffractometer (XRD-D8 Advance, Bruker Inc., Germany) and Cu K α source ($\lambda=0.15405$ nm) were used to characterize the crystal structure. The scanning step size was 0.02 $^{\circ}$, the diffraction signal was collected for 150 0s, and the X-ray diffraction data collection range was 10 $^{\circ}$ -80 $^{\circ}$. Using step scan mode, the step width is 0.01.

2.2 Statistical analysis

The data was analyzed using SPSS24.0 software (IBM, Armonk, NY, USA). Kolmogorov-Smirnov and Shapiro-Wilk methods were used to test the normal distribution of the data. One-way analysis of variance was used for statistical analysis. When $p < 0.05$, the results were considered statistically significant.

3. Results and discussion

After simulated clinical adjustment and grinding, the surface roughness of Y-TZP ceramic blocks of four brands was at the same level, and the Ra value had no significant difference ($P > 0.05$). Roughness test results show that the roughness of zirconia polished by TOB system (0.83 ± 0.10 -- $1.12 \pm 0.13 \mu\text{m}$) was the highest in other systems (0.18 ± 0.06 -- $0.65 \pm 0.03 \mu\text{m}$) ($P < 0.05$) (Tab. 2). There was considerable distinction between TOB system and control samples ($P < 0.05$). Nevertheless, the roughness of smooth samples from diverse polishing procedures were quite divergent from the control ($P < 0.05$). However, the average roughness of the monolithic zirconia after polishing by EVE system was significantly lower than that of GB, SF and KOMET ($p < 0.05$). The ordinal of roughness from high to low was TOB > GB > baseline > SF > KOMET > EVE (Tab. 2). SEM evaluation confirmed that the adjustment and polishing process altered the morphology of appearing. After grinding and polishing, the samples of each group showed apparent scratches. The zirconium blocks was polished with the EVE polishing kit (group d) with shallow surface scratches and shallow groove depths, and their number was less than the number of other groups. The number of scratches and grooves on the surface of group c was slightly larger than that of group d, but the depth and number were less than those of other groups except group d. After polishing by the TOB polishing kit (group e), the surface of the samples showed a dot-like depression, and the pores were not completely flattened, and an extensive quantity of scrapes and grooves were observed, and their width was wide. It showed that the polishing effect of TOB system was poor (Fig. 1,2). The three-dimensional surface topography of zirconia blocks showed that the surface roughness of group B (GB group) and group E (TOB group) showed obvious gully like uneven shape, more and more sharp mountain like protrusions, uneven surface color system crisscross, color span change was obvious. The surface color of group D (EVE group) was more uniform than that of other groups, with no obvious groove and peak like protrusion, and the surface was more flat. The three-dimensional morphology was consistent with the surface roughness observed by electron microscope (Fig. 3, 4).

Table 2: Roughness of surface of 4 labels of zirconium oxide after they had been modified and polished

group	Sagemax		Upcera		Cercon			Audental
baseline	0.63	± 0.06	0.92	± 0.11	0.60	± 0.15	0.71	± 0.08
GB	0.93	± 0.14	0.96	± 0.14	0.87	± 0.27	1.09	± 0.16
KOMET	0.40	± 0.05	0.46	± 0.05	0.37	± 0.09	0.43	± 0.15
EVE	0.34	± 0.03	0.32	± 0.15	0.18	± 0.06	0.32	± 0.02
TOB	0.83	± 0.10	0.89	± 0.10	0.91	± 0.07	1.12	± 0.13
SF	0.46	± 0.09	0.64	± 0.05	0.47	± 0.13	0.65	± 0.03

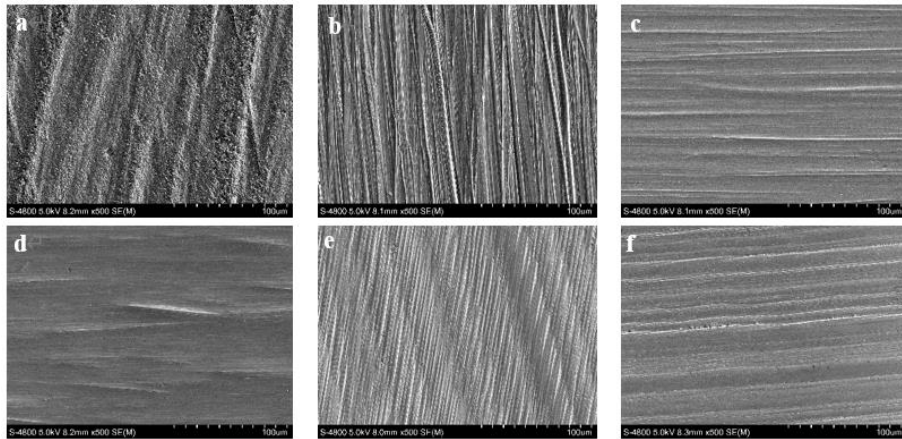


Figure 1: SEM of Upcera's full-contour zirconia after becoming subjected to various polishing procedures, a) basal line, b) GB, c) KOMET, d) EVE, e) TOB, and f) SF

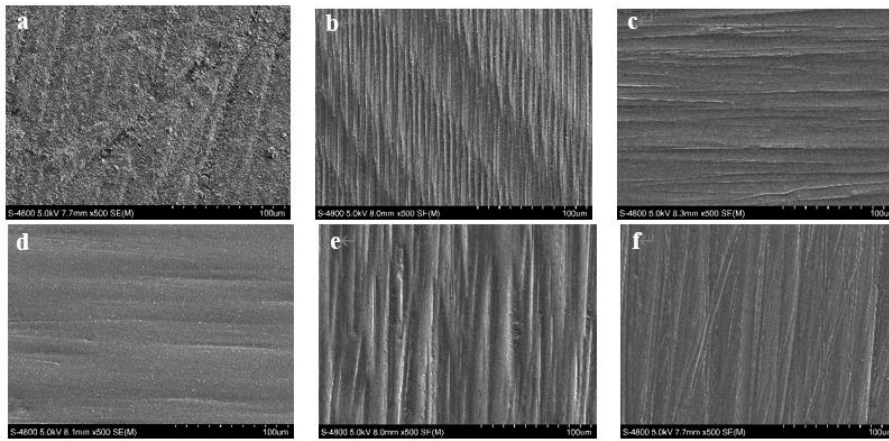


Figure 2: SEM image of Audental's full-contour zirconia after various polishing procedures, a) basal line, b) GB, c) KOMET, d) EVE, e) TOB, and f) SF

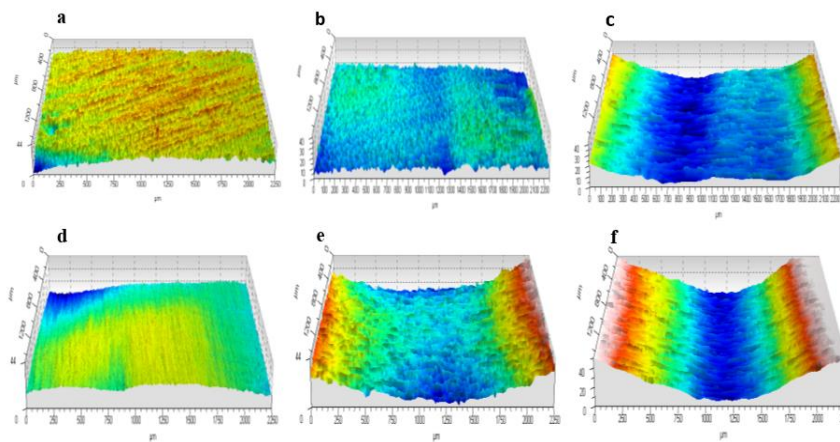


Figure 3: Three-dimensional topography of the full-contour zirconia of Upcera after being dealt with the various polishing procedures, a) basal line, b) GB, c) KOMET, d) EVE, e) TOB, and f) SF

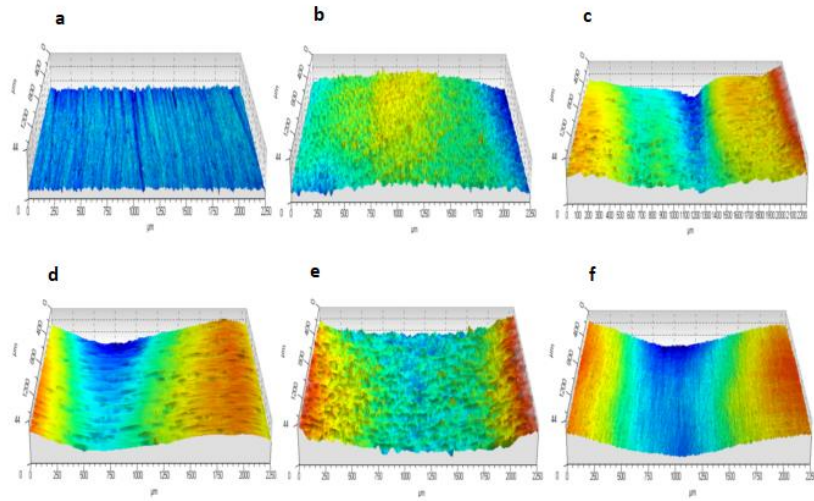


Figure 4: Three-dimensional topography of the full-contour zirconia of Audental after adjusting and polishing treatment with the various polishing procedures, a) basal line, b) GB, c) KOMET, d) EVE, e) TOB, and f) SF

The abrasive particle size of EVE coarse polishing bur was 30~50 μm . The abrasive particle shape was sharp and evenly distributed, and the local rubber binder was elongated. The abrasive particle size of EVE fine polishing bur was 3~5 μm , and the abrasive particle was well combined with the binder, and the abrasive particle fell off very little, and the rubber binder was elongated. The abrasive particle size of TOB coarse polishing bur was 20~50 μm , and the combination of abrasive particle and binder was poor, and the abrasive particle fell off more. The abrasive particle size of TOB fine polishing bur was 4~10 μm , and the abrasive particle was not tightly combined with the rubber binder (Fig.5).

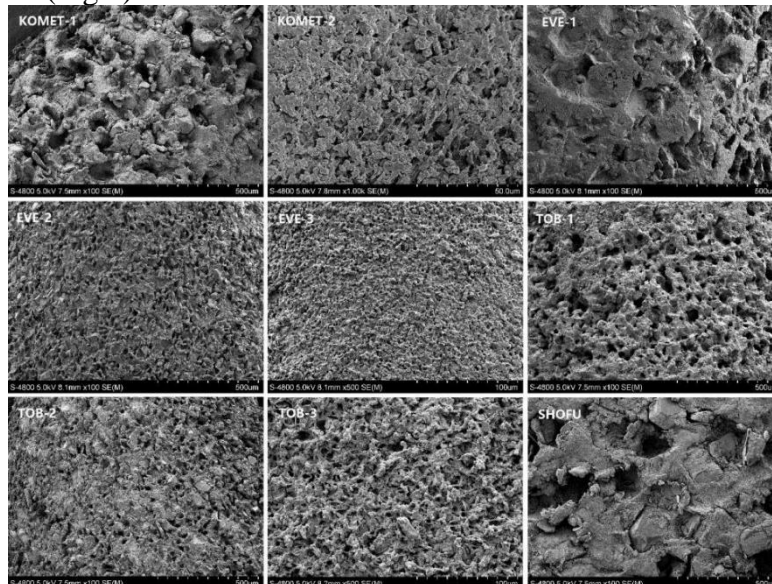
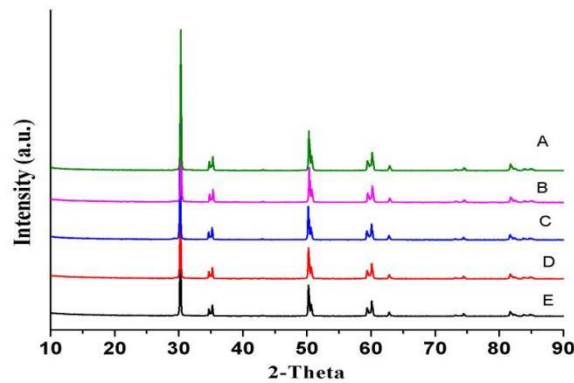


Figure 5: Scanning electron microscopy of the polishing bur. Komet-1, KOMET-2: KOMET coarse polishing bur, KOMET fine polishing bur; EVE-1, EVE-2, EVE-3: EVE coarse polishing bur, EVE fine polishing bur; TOB-1, TOB-2, TOB-3: TOB coarse polishing bur, TOB fine polishing bur; SHOFU: the SHOFU grinding head.

The XRD results are shown in Fig.6. Each group exhibited a comparable XRD pattern. The

statistics showed that the pretreatment of simulated occlusal modification and a sequence of polishing handling did not induce phase transition within zirconia samples. The tetragonal phase was the major phase for all tested samples, and no monoclinic phase was observed in any of the samples. The sharp T peaks observed in the samples revealed the significant crystallinity of tetragonal phase, and the strongest diffraction peaks obtained in XRD figure were at $2\theta = 30.232^\circ$. Further scanning were performed between 28° and 33° ; the grinding and polishing brought about peak shifts for the specimen of Weiland at baseline, GB, KOMET, EVE and SF demonstrated a crest alteration to the left, whilst TOB established a right crest transfer on the 2θ scale.



A: Polished Sagemax B: Cercon original group, C: Polished Audentalt, D: Polished Upcera, E: polished Cercon

Figure 6: XRD plots of specimens before, after adjustment and polishing procedures exhibiting peak transfers following the 2-theta axis, denoting the material's aphase transition.

Contrasted to the control samples of four labels of zirconia, XPS study demonstrated that the atomic percentage of yttrium and zirconium increased after polishing. The yttrium atomic percentage in the Cercon sample was the greatest of any specimen around polishing. Polishing afterwards, the oxygen atomic percentage in the samples of Upcera as well as Cercon were diminished. In contrast, the oxygen atomic percents in the Weiland and Diazir samples improved processing polishing. (Fig 7).

Name	Peak BE	FWHM eV	Atomic %
Y3d	157.31	3.49	1.69
Zr3d	182.33	1.81	17.52
C1s	284.73	1.66	36.47
O1s	529.61	1.42	44.43

Name	Peak BE	FWHM eV	Atomic %
Y3d	158.45	2.16	4.91
Zr3d	181.46	1.49	45.62
C1s	284.43	1.38	13.21
O1s	529.39	1.49	38.93

a) Before polishing Completed

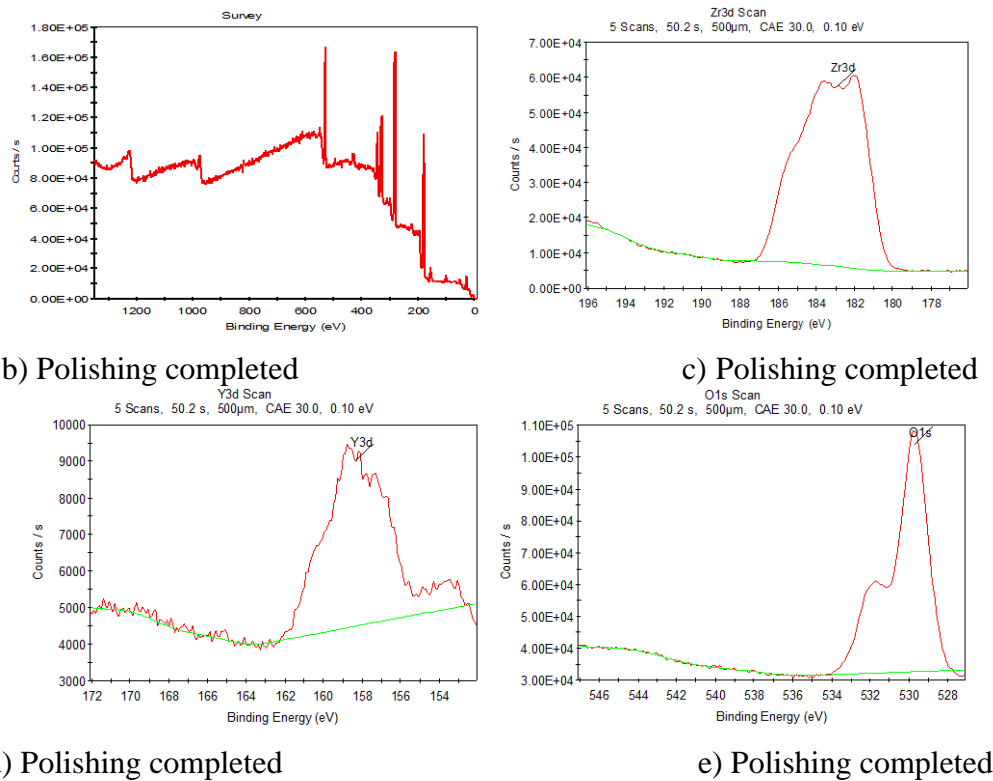


Figure 7 a-e: The element XPS and atomic ratio rates were revealed on Ucpcera zirconia specimens at the basal line, and polishing was completed.

With the application of monolithic zirconia restorations, a highly smooth Y-TZP ceramic surface has become very important, because compared to traditional porcelain crowns or all ceramic crowns with decorative porcelain, after adjusting the contour and bite, it often leads to the loss of the surface, resulting in a rough surface. The wear of the antagonist teeth is closely related to the surface roughness of the restoration, but not directly related to the hardness of the restoration [19], Low surface roughness shows less enamel wear in the matched teeth [20]. Studies have shown that surfaces with high roughness accelerate the aging of Y-TZP ceramic restorations, while polishing reduces the sensitivity of Y-TZP ceramic materials to aging [13].

Polishing can minimize the sensitivity to aging, which can be enhanced quickened by the high roughness of the zirconia. The zirconia surface has a polycrystalline structure, and the opacity increases as the surface roughness increases due to the scattering effect. Increasing opacity can reduce the aesthetics of the prosthesis. Therefore, proper surface polishing of the monolithic zirconia prosthesis is of great significance for its durative achievement. The limitation of early investigation was that simply one brand of zirconia had been experimented. However, distinct brands of zirconia may have diverse grain sizes, that can influence their transition toughness. Differentiation from early researches, 4 zirconia brands were tested in this study, and the test results of zirconia from different brands were compared, and further extensive and reasonable outcome and opinions were obtained. The 4 brands of zirconium specimens were adjusted and polished, that were mimicked the clinical handling in the trail. The various polishing systems were handled in polishing zirconium blocks by the same dentist. The impacts of diverse polishing handling on surface topography, ingredient content rate and phase variation of zirconium block around polishing were contrasted. The results showed that for monolithic zirconia crown, the faults and roughness after adjustment together with burnishing were fewer than that of all-ceramic crowns, and the roughness after polishing was lower than that was after glazing[21,22]. The polishing tools used in

this study were all special polishing tools for the monolithic zirconia, and the abrasive grain hardness on the surface was larger than the abrasive grain hardness on the surface of the polished object. Roughness assessments indicated that the EVE group had the smallest average surface roughness after polishing, succeeded by the Komet group. Compared with the baseline roughness value before polishing, the roughness of the TOB group was the highest after treatment, and it was statistically meaningful ($P < 0.05$). The place of roughness from massive to slight was that: TOB > GB > Baseline > SF > KOMET > EVE (Tab 1). It shows that EVE group had the best polishing effect, which was better than other groups. The electron micrographs and three-dimensional surface topography of zirconia blocks showed that adjustment and polishing operation altered the surface topographic characterization (Fig. 1, 2, 3, 4). Grinding with a diamond bur to create parallel depth scratches along the direction of the bur movement on the surface of the zirconia samples. This is confirmed by the SEM analysis that the gradual smoothing of the surface after using polishing Systems. All polished surfaces exhibited similar SEM images. After the EVE group of polishing, the polished zirconium block had fewer scrapes and grooves than the other groupings. (Fig. 1, 3). The outcomes were associated with the SEM of the polishing bur, that the EVE polishing kit was separated into 3 levels burs, the transition of abrasive particle size and shape among the three graded grinding burs were reasonable, the scratch texture created by the upper-level bur can be smoothed or removed by the next grinding head (Fig. 5). The transition between the grinding burs of TOB polishing kit was not since fair as the EVE kit, and the continuum was less efficient than the three-level polishing process; accordingly, the effect was poorer than the EVE kit. In order to polish the rough surfaces, the Zirconia polishing system making up of grades 2 to 4 must be applied. Average roughness after polishing should be smaller than or similar to the threshold Ra (0.2 mm) of surface of prosthesis to prevent plaque accumulation [23]. Thus, the polishing tools used in this study were all special polishing tools for monolithic zirconia, its surface hardness of diamond, silicon carbide and other abrasive grains was significantly higher than that of zirconia, which can effectively polish the occlusal of the monolithic zirconia.

When the burnish heat achieved 300 °C in the environment next persisted for 5 h, XRD diffraction discovered an m-t conversion on the superficial of zirconia, and the m phase on the zirconia sample's surface was undetectable [24]. In the study, in the adjustment period, the zirconia was grinded under the spray of water coolant to prevent the reverse phase conversion of the zirconia crystal. When compared to additional surface treatment techniques, the adjustment handling utilizing a fine diamond bit or diamond-impregnated polishing bur was gentler and had a comparatively brief period, that could decrease locally generated heat. et al [25, 26], and revealed that adjusting and other polishing practices did not proceed in an m-t conversion in the surface of the zirconia monolith. While, Pfefferle R et al. commented [27] that after polishing Y-TZP ceramics with a small particle fine grinding head and multistage polishing, no monoclinic phase was observed. Contrasted with alternative surface procedures, the grinding process with fine diamond bits or diamond impregnated polishing tools was gentler, lasted less time furthermore reduced the heat generated locally. Therefore, as previously mentioned, neither grinding nor polishing occurred in the phase transition of the zirconia detected in this research. Thus, the polishing operations possible be innocent to the zirconia phase transitions examination (Fig. 6).

From this XPS results, the Zr3d and O1s picks was very high, It showed that zirconia had good crystallinity (Fig. 7). The XPS analysis confirmed that the oxygen concentration of the Cercon and Upcera zirconia ceramics' surfaces had cut down (diminished to 38.7% , 38.93 nuclear %), the contents of yttrium of the surfaces of the Cercon- and Upcera-brand zirconia also additionally modified (increased by 3.04 and 3.22 atomic%).

While the oxygen concentration that of Sagemax- and Audental-brand zirconia ceramics had been drastically (increased to 39.78 and 39.07 atomic %), reduce in the oxygen used to be no great

of the Sagemax- and Audental-brand zirconia ceramic surfaces shows that there used to be no great yttrium precipitation the polishing and grinding measure. Although that of Sagemax- and Audental-brand zirconia also additionally improved (increased through 3.29 and 3.61 Atomic %), the expand used to be increase than that of Cercon and Upcera zirconia ceramics. This end result confirmed that in the outermost layer of Sagemax- and Audental-brand zirconia oxide, yttrium was really precipitated from the zirconia lattice and may additionally structure section of Y_2O_3 or $Y(OH)_3$. This formation made for the separation of Y_2O_3 or $Y(OH)_3$ in outermost layers and can also additionally depletion of yttrium in the localized zones of the external face. Because the yttrium concentration within those areas possibly less than the minimum for zirconia with a cubic tetragonal phase, monoclinic phases may form within those small microregions, this may additionally have an effect on the balance of the tetragonal structure of zirconia ceramics. The results showed that yttrium used to be now not uniformly disbursed in zirconia surface or body, furthermore, the yttrium and zirconium composition of various zirconia manufacturers varied. As a result of the grinding and polishing procedures, the zirconium, yttrium, carbon, and oxygen content of such zirconia surface as well as surrounding areas varies significantly, which will have an effect on the microstructure and structural balance of zirconia and additionally adversely have an effect on it. Cercon and Upcera ceramics usually have a more consistent form than the other two brands. However, the content of yttrium before and after polishing was higher than that of Upcera ceramics, so the structure properties of Cercon ceramics have been better.

There are some limitations to the study. The clinical scheme of grinding and polishing the monolithic zirconia needs to be further optimized. Due to the pressure on the handle and the cutting efficiency of burrs, more strict grinding conditions should be considered. The effects of grinding and polishing procedures on the physical and mechanical properties, reliability and clinical life of monolithic zirconia prostheses also need further researcher.

4. Conclusion

Grinding considerably improves the roughness of of 4 manufacturers of zirconia, and appropriate polishing can result in the surface of the restoration smooth to the same effect as glazed.

After the grinding and polishing, no phase transformation came about on the outermost layer of the full zirconia sample. XPS evaluation confirmed that the atomic content material of yttrium and the distribution of yttrium in 4 distinctive manufacturers of zirconia specimens had been different, which led to the alternate of the outermost layer microstructure of zirconia throughout grinding and polishing, this confirmed that this sequence of operations had certain outcomes of the performance of zirconia. Cercon zirconia had stabler structural properties.

By Comparing with different polishing systems, the EVE system produced the best polishing effect on 4 manufacturers of zirconia, while TOB kit resulted in the roughest surface morphology.

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