

2020

Effect of Artificial Aging on Hardness and Surface Roughness of Two Types of Zirconia

Ramy Hamed Elsherif

post-graduate student at Future University in Egypt, 20162685@fue.edu.eg

Mona Hossam EL-Din Mandour

Faculty of Dental Medicine for Girls, Al-Azhar University.

Rania A. Amin

Faculty of Dental Medicine for Girls, Al-Azhar University

Amr El Etreby

Faculty of Dentistry, Ain Shams University

Follow this and additional works at: <https://digitalcommons.aaru.edu.jo/fdj>



Part of the [Prosthodontics and Prosthodontology Commons](#)

Recommended Citation

Elsherif, Ramy Hamed; Mandour, Mona Hossam EL-Din; Amin, Rania A.; and El Etreby, Amr (2020) "Effect of Artificial Aging on Hardness and Surface Roughness of Two Types of Zirconia," *Future Dental Journal*: Vol. 6 : Iss. 1 , PP -.

Available at: <https://digitalcommons.aaru.edu.jo/fdj/vol6/iss1/1>

This Article is brought to you for free and open access by Arab Journals Platform. It has been accepted for inclusion in Future Dental Journal by an authorized editor. The journal is hosted on [Digital Commons](#), an Elsevier platform. For more information, please contact rakan@aarj.edu.jo, marah@aarj.edu.jo, u.murad@aarj.edu.jo.

Effect of Artificial Aging on Hardness and Surface Roughness of Two Types of Zirconia

Ramy Hamed Elsherif, Rania A Amin, Amr El Etreby

Faculty of Dental Medicine for Girls, Faculty of Dentistry, Future University in Egypt), Mona Hossam EL-Din Mandour, Al-Azhar University.), Al-Azhar University, Ain Shams University)

Abstract

Statement of the Problem: Since the developing of zirconia for dental use, aging was found to have a detrimental effect on their mechanical properties; leading to impaired function or even catastrophic failure. With the introduction of the cubic zirconia to the dental field, the effect of aging on their mechanical properties is still unclear.

Aim of the Study: This study was carried out to evaluate the effect of artificial aging (LTD) on tetragonal (3Y-TZP) and cubic (5Y-TZP) zirconia ceramics regarding hardness and surface roughness.

Materials and Methods: A total of forty discs of two brands of CAD/CAM zirconia ceramics were used in the current in-vitro study. The specimens were divided into two main groups according to the type of zirconia; Group 1 (n=20): Tetragonal zirconia (BioZX2color) and Group 2 (n=20): Cubic zirconia (DD Cube X2 98color). The required shape of the specimens was designed using digital software system in order to accurately design a cylinder shape (15mm diameter×25mm thickness) from the zirconia blanks (98mm diameter×25 mm thickness). Isomet was used to cut forty discs from their respective cylinder with dimensions (15 mm diameter × 1.5 mm thickness) which is approximately 20-25% oversize to compensate for sintering shrinkage. Then all discs were sintered, finished and polished according to manufacturer instructions.

Results: The results of surface hardness revealed that regarding both tetragonal and cubic zirconia; there was a statistically significant decrease in mean hardness after aging. The result of surface roughness revealed that regarding both tetragonal and cubic zirconia; there was a statistically significant increase in mean Ra after aging. Moreover before aging; tetragonal zirconia demonstrated a higher mean Ra which is statistically significant than cubic zirconia while, after aging; there was no statistically significant difference between mean Ra of the two zirconia types.

Conclusion: Artificial aging (low thermal degradation) negatively affects the hardness of both tetragonal and cubic zirconia. Artificial aging causes surface roughness increase for both tetragonal and cubic zirconia.

Keywords: Artificial Aging, Hardness, Surface Roughness, Zirconia

1. INTRODUCTION

The rising of zirconia (ZrO_2) as a high-execution ceramic has its sources in old paper by Garvie et al. (1975) ⁽¹⁾, and following result of others in the materials science community ⁽²⁾. It has developed into multiple variants, based on powder type, sintering substances, thermal

therapy, and other handling agents.

Of the different dopants applied, yttria (Y_2O_3) has showed to be the most effectiveness in granting an integration of high hardness and durability. So, 3 mol% (5.2 wt%) yttria settled tetragonal zirconia polycrystal (3Y-TZP) has been the pin dental ceramic for artificial repairs ⁽³⁾.

Nevertheless, classic 3Y-settled zirconia that has been proved to have excellent mechanical advantages which does not suitable for the best look demands as it is poorly translucent because it is an opaque substance. So super-translucent (ST) zirconia and cubic ultra-translucent (UT) were recently introduced. UT and ST zirconia represent a significant advancement in aesthetic monolithic CAD/CAM restorations ⁽⁴⁾.

However, zirconia ceramics have an advantage to tolerate low temperature degradation (LTD) in the dampness presence ⁽⁵⁾. This is a dynamic process in which the polycrystalline tetragonal substance gradually changes into monoclinic zirconia over a rather somewhat restrict but significant temperature range, usually between room temperature and roughly 400°C. This stage changing is tracked by micro-cracking and decaying of mechanical and physical properties of the substance ⁽⁵⁾.

Prosthetic accelerated aging is strategy which enhances the clinical state

to which substances are applied, permitting the dynamic advantages of ceramic renovation over a limited period which has been set ⁽⁶⁾.

The solidness of Y-TZP can be influenced by old age and it is a significant mechanical character to be taken in consideration in any dental procedure, it is considered to be an important marker for the substance response under padding ⁽⁷⁾.

Furthermore, LTD reinforces the loss of little surface zirconia grains in the embracing environment resulting in an increase in surface hardness and surface elevations with both aesthetic and dynamic decay. The inverse states of the mouth circumference may lead to transformations in the physical and kinetic characters of the substance because of the low temperature degradation (aging) ⁽⁸⁾.

Alghazzawi et al.(2012)⁽⁹⁾ elaborated the effect of environmental presentation to low temperature on the mechanical characters and physical firmness of dental zirconia. LTD therapy caused raised surface solidness and monoclinic stage fractions, with accompanying decrease in solidness.

There is a lack of information regarding the effect of artificial aging on the recently introduced

cubic translucent zirconia ceramic in comparison to traditional tetragonal zirconia.

The null hypotheses proposed for the present study were that artificial aging will affect neither hardness nor surface roughness of tetragonal and cubic zirconia.

2. MATERIALS AND METHODS

2.1. Materials:

The materials used, their specifications, compositions, and manufactures are listed in table (1).

3. Study design:

- A total of forty discs (N=40) of two brands of CAD/CAM zirconia ceramics were used in this in-vitro study. The sample were divided to two main groups depending on the type of zirconia: Group (1): Tetragonal Zirconia*, (n=20), Group (2): Cubic Zirconia**, (n=20).

- All specimens were subjected to the following tests: Micro-Hardness determination using Vickers hardness test, surface Roughness determination using optical non-contact profilometer, one specimen from each group was tested for change in surface morphology using Environmental Scanning Electron Microscope (ESEM).

- All specimens were subjected to an aging procedure low thermal degradation (LTD) using an autoclave (134°C, 2 bar pressure) for 5 hours.

- The same testing procedures were repeated for all specimens after LTD.

Study design is illustrated in figure (1).

4. Specimens' preparation:

Twenty Tetragonal and twenty Cubic zirconia disc shaped (N=20+20) specimens (12 mm diameter- 1.2 mm thickness) were obtained by sectioning the 2 types of zirconia blanks according to the following procedures.

4.1. Milling of zirconia blanks

The blanks were inserted inside the milling machine****, and then milled according to the imported design. The blanks were milled with an

Table 1: 1: Brand names, composition, manufacturers of the materials used in this study⁽¹⁰⁾:

Materials	Brand names	Composition	Manufacturers	Shade	Batch Number
Tetragonal Zirconia	DD BioZX ² color	3Y-TZP Yttrium stabilized Zirconia $ZrO_2 + HfO_2 + Y_2O_3 \geq 99\%$ $Y_2O_3 < 6\%$ $Al_2O_3 \leq 0.15\%$ Other oxides $< 1\%$	Dental Direkt materials Germany	A3.5	6161832007
Cubic Zirconia	DD cubeX ² 98 color	5Y-TZP Translucent Cubic Zirconia $ZrO_2 + HfO_2 \geq 99\%$ $Y_2O_3 < 10\%$ $Al_2O_3 < 0.01\%$ Other oxides $< 1\%$	Dental Direkt materials Germany	A3.5	8161706005

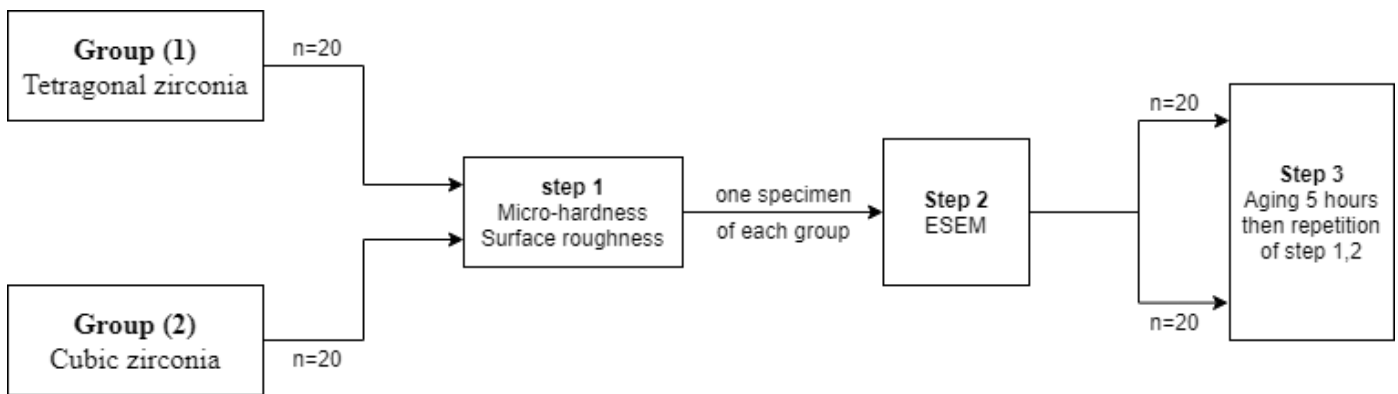


Figure 1: 1: A flowchart explaining the work flow of the study.

approximate 20-25% oversize to compensate for expected sintering shrinkage. The order to mill the blank was then given to the milling machine, after which milling was initiated.

4.2. Sectioning of the cylinders

Twenty Tetragonal and twenty Cubic zirconia disc shaped (N=20+20) specimens were then cut from their respective cylinder with dimensions (15 mm diameter × 1.5 mm thickness); to compensate for the expected sintering shrinkage, using a low speed diamond saw*, with cutting speed 2500 rpm, using diamond discs (0.7mm thickness) under cooling system considering the change of the diamond discs multiple time to assure its cutting ability throughout the whole procedure; the ratio of water coolant to the anticorrosive agent was (30:1).

4.3. Sintering of zirconia discs

The sintering process of the partially sintered zirconia specimens was conducted according to manufacturer instructions, the procedure started by placing the specimens on a sintering tray containing the appropriately sized zirconia beads, in the sintering furnace**.

The discs were completely sintered following the firing schedule illustrated in table (2) depending on the industrialist's directives for the two types of zirconia.

Table 2: 2: Standard program for zirconia sintering 1450°C /2h

	Temp. (1°C)	Temp. (2°C)	Heating Rate (°C /h)	Heating Rate (°C /min)	Dwell (°C (time/min)
Heating	20	900	480	8	
Dwell	900	900			30
Heating	900	1450	200	3	
Dwell	1450	1450			120
Cooling	1450	200	600	10	
	Total time	550 min.(9.2h)			

5. Finishing and polishing of the sintered zirconia discs

The discs were finished using ultrafine sand paper***under water coolant. Then the discs were finally polished using Eve rotary****grinding and polishing kit, with minimal pressure and under water coolant. The polishing was performed by the same operator for one surface of all the discs in the same direction and for the same number of strokes. To remove any debris contaminations all specimens were ultrasonically cleaned using Ultrasonic Cleaner***** filled with 70% ethanol for 5 minutes, and then dried using an infra-red thermal lamp*****. Digital caliper***** was used to verify the final dimensions of the discs after sintering, the final dimensions of the specimens were (12 ±0.5mm diameter × 1.2±0.25 mm thickness).

6. Artificial aging (LTD procedure):

The tested groups were subjected to Low Thermal Degradation (LTD) aging procedure in steam autoclave*****. The discs of each group were packed individually in small labeled sterilization packs which were arranged in the autoclave trays. The autoclave was programmed at 134°C, 2bars pressure for 5 hours (10 cycles) which is equivalent to the standard aging protocol to simulate oral conditions for 15 years⁽¹¹⁾. The autoclave cycle starts from zero pressure and reaches the desired pressure (2 bar) in 15 minutes so the autoclave

cycle (45 minutes) was calculated as 30 minutes. After cooling to room temperature, specimens of each group were stored in a glass tight container till testing.

7. Testing procedures

Prior and after Low Thermal Degradation (LTD) (aging procedure), the specimens were tested for the following:

All tests were conducted on the polished surface of the specimens.

7.1. Surface hardness testing

Surface toughness of the samples was detected by utilizing digital display Vickers hardness tester*, with a Vickers diamond indenter, and a 20X objective lens. A load of 300g was put to the surface of the samples for twenty seconds. For each sample the calculations of Vickers hardness number were repeated three times at variant points, the length between every point and the other was at least 0.5 m. The diagonals distances of the teething were calculated by built in scaled microscope, and Vickers values were transformed into hardness measures.

7.2. Surface Topography

The surface topography of the specimens was determined through the following:

- 1- Optical non-contact profilometer

Samples were imaged utilizing USB Digital lens with an underlying camera** attached to an IBM viable personal computer utilizing a fixed magnification of 120X. The photos were shot with a resolution of 1280×1024 pixels per photo. Digital microscope photos were edited to 350×400 pixels utilizing Microsoft office image program to indicate/limit area of hardness evaluated. The edited photos were investigated utilizing WSXM software⁽¹²⁾. By the WSXM software***, all evaluated parameters like limits, sizes and outlines are measured in pixels. So, system standardization was set transform the pixels into absolute real world units standardization was done by contrasting an object of known size (a ruler) with a scale produced by the software.

In this way, a 3D photo of the surface profile of the sample was made. Five 3D pictures were gathered for each sample, in the center at area and in the area sides of $10 \mu\text{m} \times 10 \mu\text{m}$. This area was distinguished according to the typical bacteria dimensions which is expected to adhere to restoration surface in vivo⁽¹³⁾. WSXM software was utilized to evaluate average of heights (Ra) expressed in (μm), which can be used as a real indices of surface hardness⁽¹⁴⁾.

2- Environmental Scanning Electron Microscope (ESEM):

One specimen of each group was scanned using ESEM****, before and after aging. The scanning was done to investigate any changes in the surface topography of the specimens after the LTD. The specimens were viewed at magnification power 2000X and 8000X.

8. Statistical Analysis

Numerical information was analyzed for normality by detection the distribution of information and utilizing tests of normality*. All information demonstrated normal (parametric) distribution. Information was applied as basic and standard deviation (SD) values. Two-way Analysis of Variance (ANOVA) was utilized to investigate the impact of zirconia type, aging and their interaction on mean solidness and roughness values. Bonferroni's post-hoc test was utilized for pair-

wise comparisons when ANOVA test was significant. The significance level was set at P value ≤ 0.05 . Statistical analysis was done by IBM SPSS Statistics for Windows**.

9. RESULTS

9.1. Effect of artificial aging on hardness values (VHN)

Outcomes of two-way ANOVA test showed that zirconia type regardless of aging had no statistically significant effect on mean hardness. While aging regardless of zirconia type had a statistically significant effect on mean hardness. The interaction between the variables had no statistically significant effect on mean hardness. Since the interaction between the variables being statistically non-significant, so the variables are independent from each other as shown in table (3).

9.1.1. Comparison between different variables interactions:

Whether with tetragonal or cubic zirconia; there was a statistically significant decrease in mean hardness after aging (P-value = 0.002) and (P-value = 0.027), respectively. Data are presented numerically in table (4).

10. Effect of artificial aging on surface roughness (Ra values)

10.1.

10.1.1. Statistical Analysis of surface roughness values Ra (μm)

Outcomes of two ways ANOVA showed that zirconia type, and aging had statistically significant effect on mean Ra (μm). While the interaction between the variables had no statistically significant effect on mean Ra. Since the interaction between the variables is non-statistically significant, so the variables are independent from each other. Data are presented numerically in table (5).

10.1.2. Comparison between different variables interactions

Whether with tetragonal or cubic zirconia; there was a statistically significant increase in

mean Ra (μm) after aging (P-value = 0.003) and (P-value <0.001), respectively. Data are presented numerically in table (6).

10.2. Effect of artificial aging on surface topography

10.2.1. Results of surface roughness examination

Regarding the surface topographical features, it was noticed that the surface of both tetragonal and cubic zirconia specimens after LTD, figures (3,5) revealed increased surface roughness, in addition to micro-irregularities with deeper and pointed valleys and peaks which were larger in number and pointed when compared to the surface of both tetragonal and cubic zirconia specimens before LTD, figures (2,4)

df: degrees of freedom = (n-1), *: Significant at $P \leq 0.05$, ns: non-significant

SD standard deviation, *: Significant at $P \leq 0.05$, ns: non-significant

df: degrees of freedom = (n-1), *: Significant at $P \leq 0.05$, ns: non-significant

SD standard deviation, *: Significant at $P \leq 0.05$, ns: non-significant.

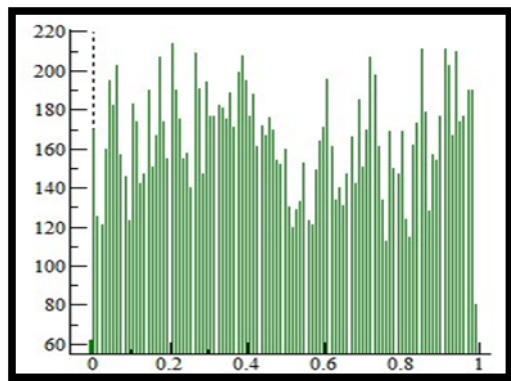


Figure 2: (b) histogram of the surface roughness.

10.2.2. Environmental Scanning Electron Microscope (ESEM examination)

The change in the surface morphology of the zirconia ceramics used in this in-vitro study was examined using ESEM.

Regarding the tetragonal zirconia; the surface of the specimen before and after LTD showed striations and cross scratches from the cutting, finishing and polishing procedures, figures (6, 7).

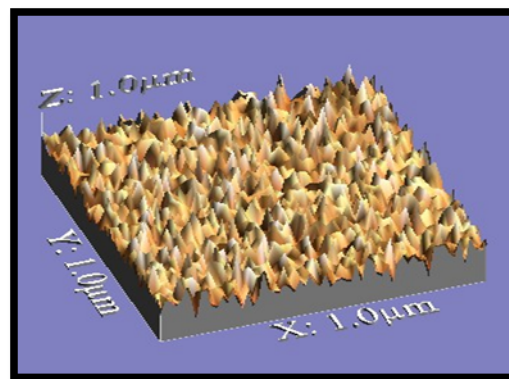


Figure 3: 2: (a) 3D image showing surface topographic features for tetragonal zirconia specimen before LTD

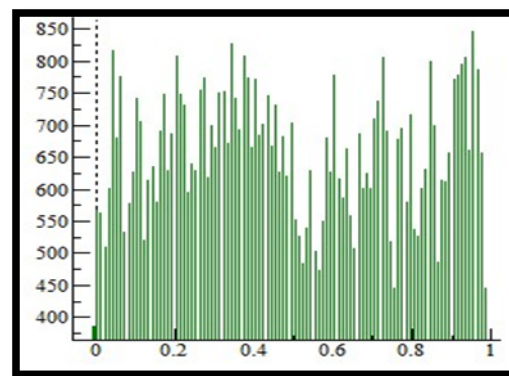


Figure 4: (b) histogram of the surface roughness.

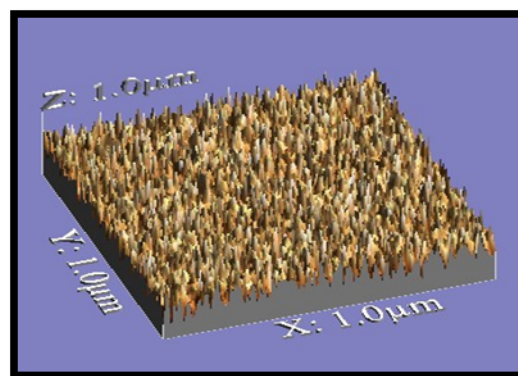


Figure 5: 3: (a) 3D image showing surface topographic features for tetragonal zirconia specimen after LTD,

Table 3: 3: Two-way ANOVA results for the effect of zirconia type, aging and their interaction on mean hardness (Kg/mm²).

Source of variation	Type III Sum of Squares	df	Mean Square	F-value	P-value
Zirconia type	6.305	1	6.305	0.008	0.930 (ns)
Aging	12965.831	1	12965.831	15.863	<0.001*
Zirconia type x Aging interaction	427.466	1	427.466	0.523	0.474 (ns)

Table 4: 4: The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between hardness values (Kg/mm²) with different interactions of variables.

Aging Groups	Before aging	SD	After aging	SD	P-value (Effect of Aging)
Group (1) Tetragonal zirconia	1175.1	43.1	1132.6	5	0.002*
Group (2) Cubic zirconia	1167.8	37	1138.3	4.7	0.027*
P-value (Effect of zirconia type)	0.570 (ns)		0.656 (ns)		

Table 5: 5: Two-way ANOVA results for the effect of zirconia type, aging and their interaction on mean Ra (μm).

Source of variation	Type III Sum of Squares	df	Mean Square	F-value	P-value
Zirconia type	0.00002	1	0.00002	5.634	0.023*
Aging	0.0001	1	0.0001	26.488	<0.001*
Zirconia type x Aging interaction	0.000001	1	0.000001	0.373	0.545 (ns)

The morphological surface of the specimen before LTD showed a clear appearance of the crystalline structure, figure (8). While after LTD, the surface of the specimen showed presence of microcracks, uplifting and irregular holes on rough surface, figures (8, 9).

Regarding the cubic zirconia; the surface of the specimen before and after LTD showed striations and scratches from the cutting, finishing and polishing procedures, figures (10, 11).

The morphologic surface of the specimen be-

fore LTD relatively showed some appearance of the crystalline structure, figure (12). While after LTD, the specimen showed presence of microcracks, and irregular holes on rough surface, in addition, there was an increase in the uplifting and irregularity along scratches after LTD, figures (12, 13).

Table 6: 6: The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between Ra (μm) values with different interactions of variables

Aging Groups	Before aging		After aging		P-value (Effect of Aging)
Group	Mean	SD	Mean	SD	
Tetragonal zirconia	0.2525	0.0013	0.2555	0.0025	0.003*
Cubic zirconia	0.2505	0.002	0.2544	0.0023	<0.001*
P-value (Effect of zirconia type)	0.042*		0.221 (ns)		

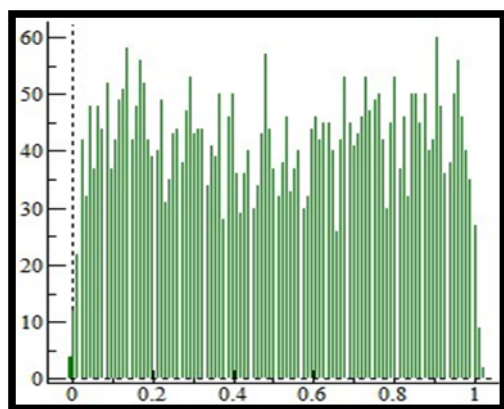


Figure 6: (b) histogram of the surface roughness.

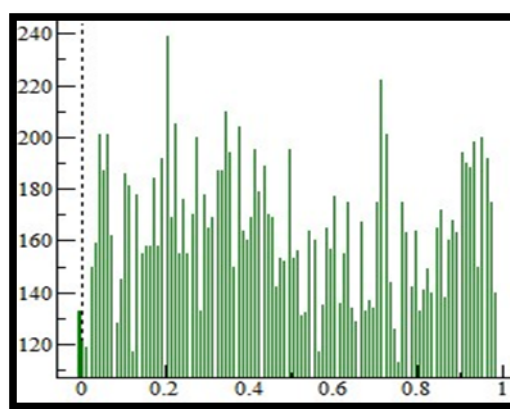


Figure 8: (b) histogram of the surface roughness.

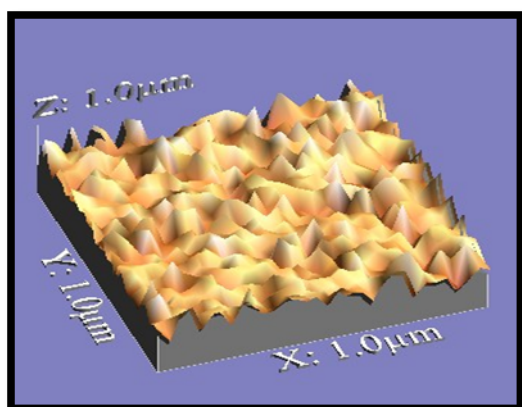


Figure 7: 4: (a) 3D image showing surface topographic features for cubic zirconia specimen before LTD

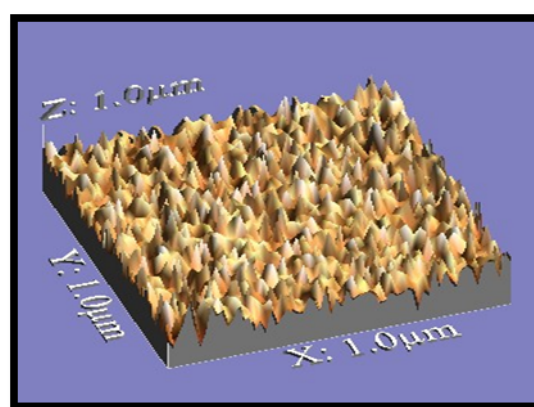


Figure 9: 5: (a) 3D image showing surface topographic features for cubic zirconia specimen after LTD

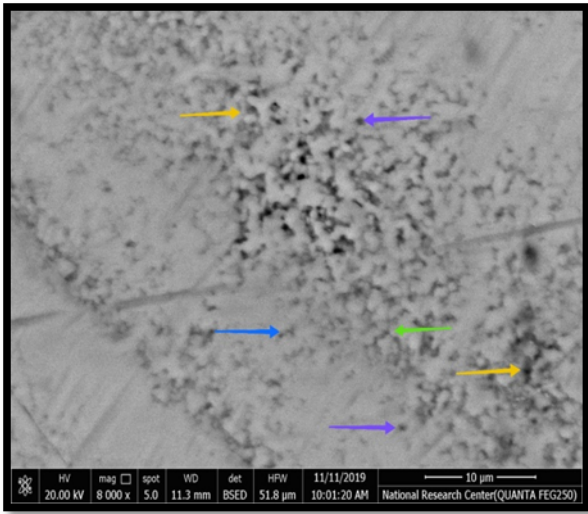


Figure 10: ESEM image for cubic zirconia specimen after LTD (8000X) showing: Uplifting and irregularities (Yellow arrows), Crystalline structure and grains (Green arrow), Micro-holes (Purple arrow) and Micro-crack (Blue arrow).

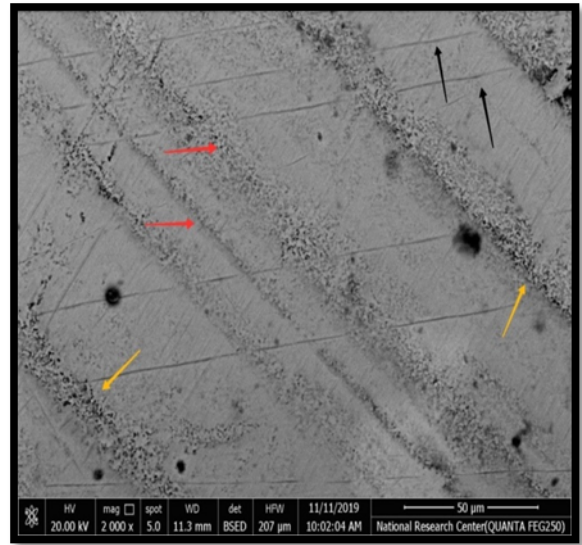


Figure 12: ESEM image for cubic zirconia specimen after LTD(2000X) showing: Striations (Red arrows), Scratches (Black arrows) and Irregularities (Yellow arrows).

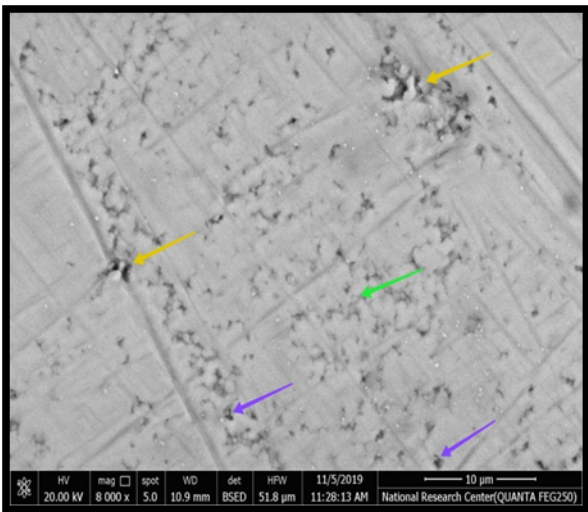


Figure 11: ESEM image for cubic zirconia specimen beforeLTD (8000X) showing: Irregularities (Yellow arrows), Crystalline structure and grains(Green arrow) and Micro-holes (purple arrow).

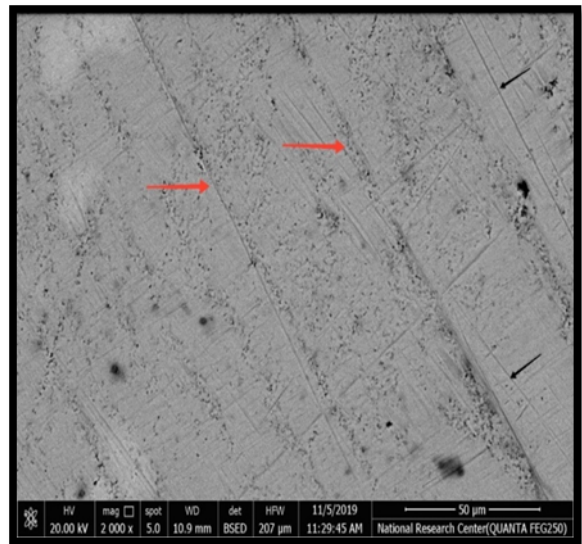


Figure 13: ESEM image for cubic zirconia specimen beforeLTD (2000X) showing: Striations (Red arrows), Scratches (Black arrows).

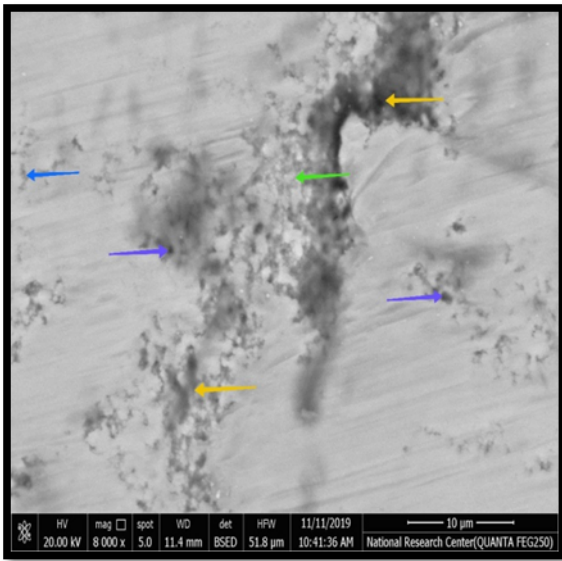


Figure 14: ESEM image for tetragonal zirconia specimen after LTD (8000X) showing: Uplifting and irregularities (Yellow arrows), Crystalline structure and grains (Green arrow), Micro-holes (Purple arrow) and Micro-crack (Blue arrow).

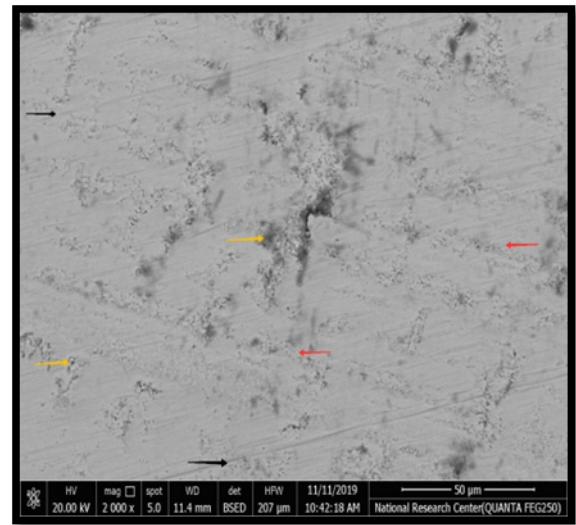


Figure 16: ESEM image for tetragonal zirconia specimen after LTD (2000X) showing: Striations (Red arrows), Scratches (Black arrows) and Irregularities (Yellow arrows).

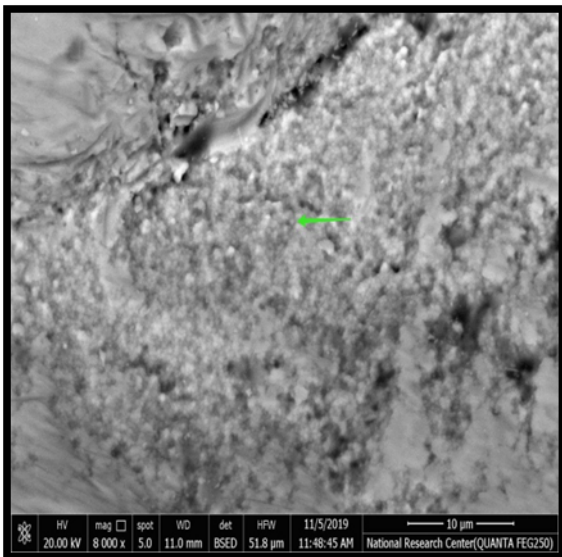


Figure 15: ESEM image for tetragonal zirconia specimen before LTD (8000X) showing: Crystalline structure (Green arrow).

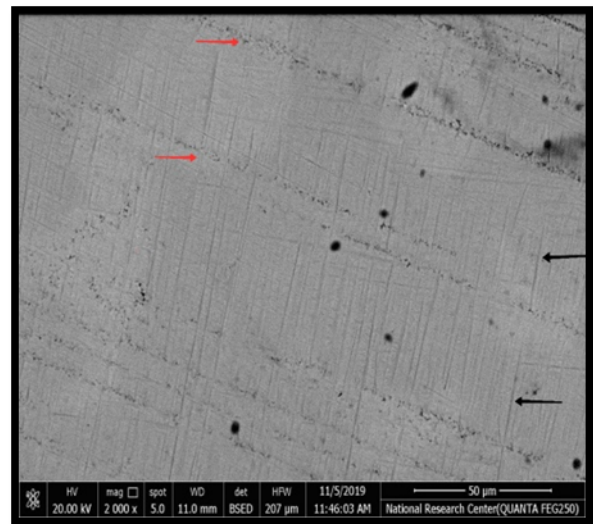


Figure 17: ESEM image for tetragonal zirconia specimen before LTD (2000X) showing: Striations (Red arrows), Scratches (Black arrows).

11. DISCUSSION

High appearance requirements of patients and optimum bio-property have led to the utilization of ceramics in dentistry restoration. Zirconia-dependent ceramics restorations are widely utilized in dentistry; nevertheless, their tendency to low temperature degradation is still difficult to achieve⁽¹⁵⁾.

In the current study, two types of CAD/CAM zirconia ceramic substances were utilized; tetragonal versus the newly introduced cubic zirconia (DD CubeX²). This material was selected as it associates the informed positive characters of zirconia with an increase in translucency significantly⁽¹⁶⁾. By adding less than 5% yttria, the tetragonal crystal stage is established to perform the classical yttria established tetragonal zirconia polycrystalline development (3Y-TZP ceramics).

The CubeX² framework is depending on a 5% yttria, which results in the formation of about 53% cubic and 47% tetragonal crystal build which vastly improves the stabilization of the new cubic zirconia 5Y-TZP molecular build⁽¹⁷⁾. As a result of the cubic phase increase, the translucency of this new material increased by 49% due to the isotopic nature of the cubic grains which improves light transmission, unlike the tetragonal grains with the anisotropic nature that leads to impaired light transmission due to light scattering⁽¹⁸⁾. However, mechanical properties were affected as cubic zirconia is not exposed to stress-prompted conversion⁽¹⁶⁾.

The majority of zirconia dental restorative substances undergo disintegration of strength measures along time as aging/stage conversion. Because of the induced establishment of the cubic stage in CubeX² in contrast with the tetragonal stage present in prosthetic zirconia products, less stage conversion is noticed in-vivo, permitting CubeX² to keep much more of its first power over time.

In the present study forty discs (12 mm diameter x 1.2 mm thickness) were cut from cylinders milled from zirconia blanks; these dimensions were selected according to ISO: 6872:2008

standards^(19,20).

Sintering of the specimens was performed according to the manufacturer's recommendation in a high temperature furnace at 1450°C according to ISO 13356:2015 to generate dense structures which ensure the cohesion between the zirconia grains, as dense material prevents penetration of the water to the bulk of the material⁽¹¹⁾.

Finishing and polishing of the specimens was performed in order to simulate the real state for the clinical situation, a standardized finishing and polishing protocol was used to ensure similar base line Ra values, followed by ultrasonic cleaning and dryness of the specimens to remove any manipulative contamination that may affect the results^(19,20). Similar base line Ra values were confirmed by roughness testing prior to aging, where there is no statistically significant difference between the two groups was detected.

In the current study low thermal degradation (LTD) aging procedure of the zirconia specimens was induced using steam autoclave, as it is an established method for accelerated aging in Y-TZP according to standard aging protocol which is equivalent to 15-year clinical conditions⁽²¹⁾. This approach is in accordance with a previous study by Lughfi and Sergo⁽⁶⁾ which considered the treatment of zirconia specimens in the autoclave for 1 hour at 134 °C to be equivalent to 3-4 years of in-vivo aging. Moreover, Sergo⁽²²⁾ reported that 5 hours aging at 134°C corresponds to 15-20 years at 37 °C.

The process of aging composed of a tetragonal to monoclinic conversion of the surface grains in relation to molecules of water⁽²³⁾. This conversion of surface is accompanied by the uplifts development on the surface and finally micro-breaking and grain pull-out, which may initiate an advanced decay of mechanical characters⁽²⁴⁾.

The most admitted theory to demonstrate mechanism of LTD is that the internal stress increases accompanied by the water (H₂O) penetration within the trellis inductions the beginning of the t-m stage conversion⁽²⁵⁾. So an event sequence happens with the conversion progress initially within one grain^(26,27), and advancing infestation the surface by a nucleation-and-growth

(N-G) technique^(23,24,28). The nuclei number increases relatively with the burdens, due to the water penetration (time dependent)⁽²⁹⁾. Simultaneously, growth happens owing to the reality that the conversion of one-grain makes its relatives under tense pressure, preferring their conversion under the water impact⁽²⁴⁾. Thus, LTD first happens at grains superficially where water is united to zirconia grains by stuffing oxygen vacancies, later surface spreading will increase its sturdiness^(30,31), and decrease the solidness⁽³²⁾. Moreover, LTD progresses into the substance bulk and endangers the strength, cracking sturdiness, and Y-TZP thickness structures^(6,33).

The t-m stage conversion spreads slowly from the surface into the mass in the LTD technique. The converted layer depth is depending on time and was evaluated to propagate a little micrometers⁽³⁴⁾. A sequence in which, processes concentrated on surface properties like solidness and surface sturdiness should be used and associated to the variations in mechanical characters after LTD⁽³²⁾⁽³⁵⁾.

Micro-hardness testing is an immediate strategy when examining the dynamic characters and was utilize to characterize the hydrothermally aged zirconia effectively^(36–38). A digital display Vickers micro-hardness tester was used to detect the micro-hardness of the tetragonal and cubic zirconia specimens before and after aging. The importance of using micro-hardness indentation test includes the ability to generate a load hardness curve of a material and thus easily compared to other materials. Moreover the qualitative information from the indentation damage can be transferred through image processing into quantitative information⁽³⁹⁾.

The outcomes of the current study demonstrated that (LTD) aging decreases the surface hardness in both tetragonal and cubic zirconia samples. These outcomes were in agreement with old studies^(37,40–45). These studies showed a powerful correlation between the decrease in surface hardness and the increase in monoclinic fraction. This may be due to the increase in volume related to the t-m t changing that results in micro-fissures formation⁽⁴¹⁾, and decreases the

local atomic density⁽⁴⁰⁾.

In the present study the outcomes reported that there was no statistical significant difference in hardness values between both the cubic zirconia and tetragonal. These data are enhanced by Shen et al.⁽⁴⁶⁾ Who studied the hydrothermal impact disintegration on 5Y-TZP and 3Y-TZP, and deduced that there was no statistical variation in surface sturdiness between two types of zirconia. Which may be explained by fact of differences in yttria structure and crystal stages, there are no many contrasts between any zirconia type in the versatile modulus, Vickers hardness, or the heat expansion coefficient. This recommends that these characters are depending on Zr and O bonding strength strongly, and not depending on the crystal phase fraction. So, chemical characters like degradation resistance are approximately the same which is strongly based on the crystal stage and changes with the yttria structure radically⁽⁴⁷⁾.

On the disagreement to our results of this study Moqbel et al.⁽⁴⁸⁾ who studied the impact of aging on Vickers hardness (VHN) of translucent dental zirconia utilizing an autoclave for 20 h (134°C and 0.2 MPa) and they reported that hardness was affected by aging. The reason for this may be due to the transformation percentage of t-m phase was not enough to initiate surface deterioration resulting in same hardness values as before aging.

The optical non-contact profilometer was used to measure the surface roughness and provides topographic 3D images of the tested zirconia ceramics before and after aging due to: it's easier access, reliability and affordability with a great degree of accuracy, it has being used successfully in lot of studies^(49–51). Unlike other technology, it does not require either vacuum or sample treatment that might cause damage^(12–14), it gives a quantitative aspect through calculation of the difference between the depth of two different points in the surface, which cannot be obtained with the SEM⁽⁵²⁾, the white light confocal laser together with the utilize of non-contact version allowing precise evaluation of the zirconia surface roughness samples⁽⁵³⁾, non-contact optical profilometer avoid the limitation of the tip diameter of the stylus found in the stylus profilometer which faced

the problem of being large than the grain size of zirconia so giving inaccurate results⁽⁴³⁾.

Besides its deleterious effect on the aesthetic appearance and wear of the antagonist⁽⁵³⁾, the importance to evaluate zirconia roughness is due to the fact that the oral biofilm tends to grow more on rough surfaces⁽⁵⁴⁾. Also, bacteria naturally tend to adhere to areas protected from mechanical action⁽⁵⁵⁾. Thus, if there is doubt whether aging can increase the material's roughness in the long term (which can lead to more microorganism adhesion), it should be evaluated⁽⁵⁶⁾.

In the present study, statistical results revealed that aging significantly increases the surface roughness for the tested tetragonal and cubic zirconia ceramics. This could be due to the fact that at low temperature range between 125°C and 150°C (which is the range of current aging test) transformation from tetragonal to monoclinic procedure begin to proceed rapidly with uplifting of some grains, pushing them out to the surface causing micro-fissures that as result will open the property for water penetration beneath the surface, so spreading the t-m changing to the interior of the specimen⁽⁵⁷⁾, and eventually, it will result in the formation of large fissures leading to surface roughness⁽⁵⁸⁾.

These results were supported by the morphologic scan of the specimens which showed the presence of multiple irregular holes and internal micro-cracks which gives an impression about increase in the surface roughness after LTD. These results were predictable for hybrid tetragonal-cubic zirconia, as when cubic grains exist with tetragonal grains, LTD is accelerated as cubic grains attract yttria from relatively tetragonal grains, influencing its stabilization^(59,60).

These results were in agreement with previous studies^(22,61) who found that surface roughness of the zirconia increased after being exposed to hydrothermal aging at low temperature when aging procedure performed in an autoclave at 134°C at 2 bars for 3hours⁽²²⁾.

On the contrary to our findings Amaral et al.⁽⁵¹⁾ concluded that aging did not promote any relevant surface change. They explained that through the fact that samples were mirror-

polished, thus after LTD samples might retain its polishing surface. This aging schedule was carried out very quickly and without need to any mechanically. As a result to that, it may not lead to pull-out of grain, leading to identical roughness esteems to the non-aged zirconia⁽⁶¹⁾.

De Souza et al.⁽⁶²⁾ studied the effect of aging on surface topography and hardness of yttria-tetragonal zirconia polycrystal (Y-TZP) through artificial aging in autoclave. They found that there was an increase in surface roughness accompanied by grain pull-out which supports the finding of our current study. While there was no effect on hardness, on the contrary to the findings of this study. This might be explained through the fact that the conversion of t-m stage related to aging not only it produces a primary deformity, but also it results in increasing the size of grain. This procedure produces a regional pressure stress on the surface which resorts to shut a possibly progressing fissure to keep its mechanistic equilibrium, that may result in an increase in mechanistic characters⁽⁶⁾. Within the zirconia hydrothermal aging, monoclinic stage development produces two main effects which are, increasing in the pressure stress that rises mechanistic characters and the development of disorders, that reduces these characters⁽⁶³⁾. So, basing on the power of every impact, the substance mechanistic characters may rise, reduce or kept without any change.

Long term clinical success of dental zirconia restorations depends on a number of factors, one of these factors is the roughness, surface roughness influences the initial bacterial adhesion, rough zirconia restoration surface will accumulate more plaque due to presence of irregularities, these irregularities can serve as shelter for microorganisms, protecting them from forces of salivary flow, chewing, swallowing and oral hygiene measures; favoring microbial colonization, and possibly leading to biological and/or esthetic failure of the material^(64,65).

The decrease in hardness values results in microcracking, grain pull out and decrease in wear resistance which will be responsible for higher wear values of the enamel antagonists⁽⁶⁶⁾.

Further studies are needed using different artificial aging protocols; also In-vivo studies are needed to validate the in-vitro results and to understand the real performance of tetragonal and cubic zirconia in the oral cavity.

12. CONCLUSION

Within the limitations of the present study, the following could be concluded:

1. Artificial aging (low thermal degradation) negatively affects the hardness of both tetragonal and cubic zirconia.
- 2-Artificial aging causes surface roughness increase for both tetragonal and cubic zirconia

13. References

1. Garvie R.C, Hannink R.H, Pascoe R.T. Ceramic steel, *Int. J Nat. Sc.* 1975; 258: 703-704.
2. Green DJ, Hannink RHJ, Swain MV. Transformation toughening of ceramics. 1st ed.CRC Press, 1989.
3. Klimke J, Trunec M, Krell A. Transparent tetragonal yttria-stabilized zirconia ceramics: influence of scattering caused by birefringence. *J. Am. Ceram. Soc.* 2011; 94: 1850-1858.
4. Stawarczyk B, Özcan M, Schmutz F, Trottmann A, Roos M, Hämmerle CH. Two-body wear of monolithic, veneered and glazed zirconia and their corresponding enamel antagonists. *J. Acta. Odontol. Scand.* 2013; 71:102-120.
5. Ban S, Suehiro Y, Nakanishi H, Nawa H. Fracture toughness of dental zirconia before and after autoclaving. *J. Ceram. Soc. Jpn.* 2010; 118: 406-409.
6. Lughi V, Sergo V. Low temperature degradation -aging- of zirconia: A critical review of the relevant aspects in dentistry. *J. Dent. Mater.* 2010; 26: 807-820.
7. Ramesh S, Lee KS, Tan C. A review on the hydrothermal ageing behaviour of Y-TZP ceramics. *J. Ceram. Int.*2018;44: 20620-20634.
8. Lee TH, Lee SH, Her SB, Chang WG, Lim BS. Effects of surface treatments on the susceptibilities of low temperature degradation by autoclaving in zirconia. *J.Biomed.Mater.Res. B.* 2012; 100:1334-1343.
9. Alghazzawi TF, Lemons J, Liu PR, Essig ME, Bartolucci AA, Janowski GM. Influence of low-temperature environmental exposure on the mechanical properties and structural stability of dental zirconia. *J. Prosthodont.* 2012;21:363-369.stability of dental zirconia. 2012;21(5):363-9.
- 10.<https://www.dentaldirekt.de/en/products/materials/zirconium-dioxide>.
11. Inokoshi M, Zhang F, De Munck J, Minakuchi S, Naert I, Vleugels J, Van MB, Vanmeensel K. Influence of sintering conditions on low-temperature degradation of dental zirconia. *J. Dent. Mater.* 2014;30:669-678.
12. Horcas I, Fernández R, Gomez-Rodriguez J, Colchero J, Gómez-Herrero J, Baro A. WSXM: a software for scanning probe microscopy and a tool for nanotechnology. *J. Rev. Sci. Instrum.* 2007;78:013705.
13. Giacomelli L, Derchi G, Frustaci A, Bruno O, Covani U, Barone A, De Santis D, Chiappelli F. Surface roughness of commercial composites after different polishing protocols: an analysis with atomic force microscopy. *J. Open Dent.* 2010;4:191-194.
14. Kakaboura A, Fragouli M, Rahiotis C, Silikas N. Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and glossmeter. *J. Mater. Sci. Mater. Med.* 2007;18:155-163.
15. Zhang Y, Chen J, Hu L, Liu W. Pressureless-sintering behavior of nanocrystalline ZrO₂-Y₂O₃-Al₂O₃ system. *J. Mater. Lett.* 2006; 60:2302-2305.
16. McLaren EA, Lawson N, Choi J, Kang J, Trujillo C. New high-translucent cubic-phase-containing zirconia: clinical and laboratory considerations and the effect of air abrasion on strength. *J. Compend. Cont. Educat. Dentistry* 2017;38:13-6.
17. Elsayed A, Meyer G, Wille S, Kern M. Influence of the yttrium content on the fracture strength of monolithic zirconia crowns after artificial aging. *J. Quintessence Int.* 2019;50: 344-348.
18. Zhang YJ. Making yttria-stabilized tetragonal zirconia translucent. *J. Dent. Mater.*2014;30:1195-1203.

19. International Organization for Standards. ISO6872: Dental ceramic. Geneve. 2008.
20. Muñoz EM, Longhini D, Antonio SG, Adabo GL. The effects of mechanical and hydrothermal aging on microstructure and biaxial flexural strength of an anterior and a posterior monolithic zirconia. *J. Dent.* 2017;63:94-102.
21. Cattani-Lorente M, Durual S, Amezdroz M, Wiskott HA, Scherrer SS. Hydrothermal degradation of a 3Y-TZP translucent dental ceramic: A comparison of numerical predictions with experimental data after 2 years of aging. *J. Dent. Mater.* 2016;32:394-402.
22. Sergo V. Room-temperature aging of laminate composites of alumina/3-mol%-yttria-stabilized tetragonal zirconia polycrystals. *J. Am. Ceram. Soc.* 2004;87:247-253
23. Chevalier J, Cales B, Drouin JMJJotACS. Low-temperature aging of Y-TZP ceramics. 1999;82(8):2150-4.
24. Chevalier J, Gremillard L, Deville S. Low-temperature degradation of zirconia and implications for biomedical implants. *J. Annu. Rev. Mater. Res.* 2007;37:1-32.
25. Schubert H, Frey F. Stability of Y-TZP during hydrothermal treatment: neutron experiments and stability considerations. *J. Europ. Ceram. Soc.* 2005;25:1597-1602.
26. Deville S, Chevalier J, Fantozzi G, Bartolomé JF, Requena Jn, Moya JS, et al. Low-temperature ageing of zirconia-toughened alumina ceramics and its implication in biomedical implants. 2003;23(15):2975-82.
27. Schmauder S, Schubert H. Significance of internal stresses for the martensitic transformation in yttria-stabilized tetragonal zirconia polycrystals during degradation. *J. Am. Ceram. Soc.* 1986;69:534-540.
28. Muñoz-Tabares J, Jiménez-Piqué E, Anglada M. Subsurface evaluation of hydrothermal degradation of zirconia. *J. Acta. Mater.* 2011;59:473-484.
29. Lucas TJ, Lawson NC, Janowski GM, Burgess J. Effect of grain size on the monoclinic transformation, hardness, roughness, and modulus of aged partially stabilized zirconia. *J. Dent. Mater.* 2015;31:1487-1492.
30. Sato T, Shimada M. Transformation of yttria-doped tetragonal ZrO₂ polycrystals by annealing in water. *J. Am. Ceram. Soc.* 1985;68:356.
31. Yoshimura M, Noma T, Kawabata K, Sōmiya S. *Hydrothermal Reactions for Materials Science and Engineering*. 1st ed. Elsevier Applied Science, 1989.
32. Ardlin B. Transformation-toughened zirconia for dental inlays, crowns and bridges: chemical stability and effect of low-temperature aging on flexural strength and surface structure. *J. Dent. Mater.* 2002;18:590-595.
33. Ban S, Sato H, Suehiro Y, Nakanishi H, Nawa M. Biaxial flexure strength and low temperature degradation of Ce-TZP/Al₂O₃ nanocomposite and Y-TZP as dental restoratives. *J. Biomed. Mater. Res. B. appl. Biomater.* 2008;87:492-498.
34. Chowdhury S, Vohra YK, Lemons JE, Ueno M, Ikeda J. Accelerating aging of zirconia femoral head implants: change of surface structure and mechanical properties. *J. Biomed. Mater. Res. B. Appl. Biomater.* 2007;81:486-492.
35. Prado PHCO, Monteiro JB, Campos TMB, Thim GP, de Melo RM. Degradation kinetics of high-translucency dental zirconias: Mechanical properties and in-depth analysis of phase transformation. *J. Mech. Behav. Biomed. Mater.* 2020;102: 103482.
36. Guo X, Schober T. Water incorporation in tetragonal zirconia. *J. Am. Ceram. Soc.* 2004;87:746-748.
37. Zhang F, Inokoshi M, Batuk M, Hadermann J, Naert I, Van Meerbeek B, Vleugels J. Strength, toughness and aging stability of highly-translucent Y-TZP ceramics for dental restorations. *J. Dent. Mater.* 2016;32: 327-337.
38. Monzavi M, Zhang F, Meille S, Douillard T, Adrien J, Noubissi S, Nowzari H, Chevalier J. Influence of artificial aging on mechanical properties of commercially and non-commercially available zirconia dental implants. *J. Mech. Behav. Biomed. Mater.* 2020;101:103423.
39. Alhenaki AM. Comparison of mechanical And optical properties between three different CAD/CAM materials. Master's thesis. Nova

Southeastern University, 2015.

40. Catledge SA, Cook M, Vohra YK, Santos EM, McClenny MD, Moore KD. Surface crystalline phases and nanoindentation hardness of explanted zirconia femoral heads. *J. Mater. Sci. Mater. Med.* 2003;14:863-867.

41. Mitov G, Anastassova-Yoshida Y, Nothdurft FP, Von See C, Pospiech P. Influence of the preparation design and artificial aging on the fracture resistance of monolithic zirconia crowns. *J. Adv. Prosthodont.* 2016;8:30-36.

42. Casucci A, Mazzitelli C, Monticelli F, Toledano M, Osorio R, Osorio E, Papacchini F, Ferrari M. Morphological analysis of three zirconium oxide ceramics: Effect of surface treatments. *J. Dent. Mater.* 2010;26:751-760.

43. Nakamura K, Harada A, Ono M, Shibasaki H, Kanno T, Niwano Y, Adolfsson E, Milleding P, Örtengren U. Effect of low-temperature degradation on the mechanical and microstructural properties of tooth-colored 3Y-TZP ceramics. *J. Mech. Behav. Biomed.* 2016;53:301-311.

44. Elshazly ES, El-Hout S, Ali ME-S. Yttria tetragonal zirconia biomaterials: kinetic investigation. *J. Mater. Sci. Technol.* 2011;27:332-337.

45. Cattani-Lorente M, Scherrer SS, Ammann P, Jobin M, Wiskott HA. Low temperature degradation of a Y-TZP dental ceramic. *J. Acta. Biomater.* 2011;7:858-865.

46. Shen J, Xie H, Wu X, Yang J, Liao M, Chen C. Evaluation of the effect of low-temperature degradation on the translucency and mechanical properties of ultra-transparent 5Y-TZP ceramics. *J. Ceram. Int.* 2020;46:553-559.

47. Ban S. Chemical durability of high translucent dental zirconia. *J. Dent. mater.* 2019;39:12-23.

48. Moqbel NM, Al-Akhali M, Wille S, Kern M. Influence of aging on biaxial flexural strength and hardness of translucent 3Y-TZP. *J. Mater.* 2020;13:13-27.

49. Gremillard L, Martin L, Zych L, Crosnier E, Chevalier J, Charbouillot A, Sainsot P, Espinouse J, Aurelle, J-L. Combining ageing and wear to assess the durability of zirconia-based ceramic heads for total hip arthroplasty. *J. Acta. Biomater.* 2013;9:7545-7555.

50. Borchers L, Stiesch M, Bach F-W, Buhl J-C, Hübsch C, Kellner T, Kohorst P, Jendras M. Influence of hydrothermal and mechanical conditions on the strength of zirconia. *J. Acta. Biomater.* 2010;6:4547-4552.

51. Amaral M, Weitzel ISSL, Silvestri T, Guilardi LF, Pereira GKR, Valandro LF. Effect of grinding and aging on subcritical crack growth of a Y-TZP ceramic. *J. Braz. Oral Res.* 2018;32:00-32.

52. Innes JM. An investigation into surface texture and in-vitro two-body wear of CAD/CAM dental materials antagonised by acrylic denture teeth. Master thesis, University of Adelaide, 2016.

53. Bartolo D, Cassar G, Husain NA-H, Özcan M, Camilleri J. Effect of polishing procedures and hydrothermal aging on wear characteristics and phase transformation of zirconium dioxide. *J. Prosthet. Dent.* 2017;117:545-551.

54. Dal Piva A, Contreras L, Ribeiro F, Anami L, Camargo S, Jorge A, Bottino MA. Monolithic ceramics: effect of finishing techniques on surface properties, bacterial adhesion and cell viability. *J. Oper. Dent.* 2018;43:315-325

55. Kidd E, Fejerskov O. What constitutes dental caries? histopathology of carious enamel and dentin related to the action of cariogenic biofilms. *J. Dent. Res.* 2004;83:35-38.

56. Dal AP, Tribst JP, Gondim LD, Ribeiro IL, Campos F, Arata A, Souza RO. Y-TZP surface behavior under two different milling systems and three different accelerated aging protocols. *J. Minerva. Stomatol.* 2018;67:237-245.

57. Zhang F, Vanmeensel K, Batuk M, Hadermann J, Inokoshi M, Van Meerbeek B, Naert I, Vleugels J. Highly-translucent, strong and aging-resistant 3Y-TZP ceramics for dental restoration by grain boundary segregation. *J. Acta. Biomater.* 2015;16:215-222.

58. Matei M, Voinea E-A, Rîcă R, Manolea H, Mogoantă L, Salan A, Rîcă A, Dinescu VC, Cioateră N. New zirconia-based materials for dental applications. Structural, morphological and histological evaluation. *J. Ceram. Int.* 2019;45:14859-14866.

59. Zhu WZ. Effect of cubic phase on the kinetics of the isothermal tetragonal to monoclinic

transformation in ZrO₂ (3mol% Y₂O₃) ceramics. *J. Ceram. Int.* 1998;24:35-43.

60. Chevalier J, Deville S, Münch E, Jullian R, Lair F. Critical effect of cubic phase on aging in 3 mol% yttria-stabilized zirconia ceramics for hip replacement prosthesis. *J. Biomater.* 2004;25:5539-5545.

61. Guilardi LF, Pereira GKR, Wandscher VF, Rippe MP, Valandro LF. Mechanical behavior of yttria-stabilized tetragonal zirconia polycrystal: effects of different aging regimens. *J. Braz. Oral Res.* 2017;31:94-104.

62. De Souza GM, Zyklus A, Ghahnavyeh RR, Lawrence SK, Bahr DF. Effect of accelerated aging on dental zirconia-based materials. *J. Mech. Behav. Biomed. Mater.* 2017;65: 256-263.

63. Pereira G, Amaral M, Cesar P, Bottino M, Kleverlaan C, Valandro L. Effect of low-temperature aging on the mechanical behavior of ground Y-TZP. *J. Mech. Behav. Biomed. Mater.* 2015; 45:183-192.

64. Fais LM, Fernandes-Filho RB, Pereira-da-Silva MA, Vaz LG, Adabo GL. Titanium surface topography after brushing with fluoride and fluoride-free toothpaste simulating 10 years of use. *J. Dent.* 2012;40:265-275.

65. Scotti R, Kantorski KZ, Monaco C, Valandro LF, Ciocca L, Bottino MA. SEM evaluation of in situ early bacterial colonization on a Y-TZP ceramic: a pilot study. *J. Int. Prosthodont.* 2007;20: 419-422.

66. Aboushahba M, Katamish H, Elagroudy M. Evaluation of hardness and wear of surface treated zirconia on enamel wear. An in-vitro study, *Futur. Dent. J.* 2018;76-83.

References

- [1] R. C. GARVIE, R. H. HANNINK, R. T. PASCOE, Ceramic steel?, *Nature* 258 (5537) (1975) 703–704. doi:10.1038/258703a0. URL <https://dx.doi.org/10.1038/258703a0>
- [2] D. J. Green, R. Hannink, M. V. Swain (1989).
- [3] J. Klimke, M. Trunec, A. Krell, Transparent Tetragonal Yttria-Stabilized Zirconia Ceramics: Influence of Scattering Caused by Birefringence, *Journal of the American Ceramic Society* 94 (6) (2011) 1850–1858. doi:10.1111/j.1551-2916.2010.04322.x. URL <https://dx.doi.org/10.1111/j.1551-2916.2010.04322.x>
- [4] B. Stawarczyk, M. Özcan, F. Schmutz, A. Trottmann, M. Roos, C. H. F. Hämmerle, Two-body wear of monolithic, veneered and glazed zirconia and their corresponding enamel antagonists, *Acta Odontologica Scandinavica* 71 (1) (2013) 102–112. doi:10.3109/00016357.2011.654248. URL <https://dx.doi.org/10.3109/00016357.2011.654248>
- [5] S. BAN, Y. SUEHIRO, H. NAKANISHI, M. NAWA, Fracture toughness of dental zirconia before and after autoclaving, *Journal of the Ceramic Society of Japan* 118 (1378) (2010) 406–409. doi:10.2109/jcersj2.118.406. URL <https://dx.doi.org/10.2109/jcersj2.118.406>
- [6] V. Lughì, V. Sergo, Low temperature degradation -aging- of zirconia: A critical review of the relevant aspects in dentistry, *Dental Materials* 26 (8) (2010) 807–820. doi:10.1016/j.dental.2010.04.006. URL <https://dx.doi.org/10.1016/j.dental.2010.04.006>
- [7] S. Ramesh, K. Y. S. Lee, C. Y. Tan, A review on the hydrothermal ageing behaviour of Y-TZP ceramics, *Ceramics International* 44 (17) (2018) 20620–20634. doi:10.1016/j.ceramint.2018.08.216. URL <https://dx.doi.org/10.1016/j.ceramint.2018.08.216>
- [8] T. H. Lee, S. H. Lee, S. B. Her, W. G. Chang, B. S. Lim, Effects of surface treatments on the susceptibilities of low temperature degradation by autoclaving in zirconia, *J. Biomed. Mater. Res. B* 100 (2012) 1334–1343.
- [9] T. F. Alghazzawi, J. Lemons, P.-R. Liu, M. E. Essig, A. A. Bartolucci, G. M. Janowski, Influence of Low-Temperature Environmental Exposure on the Mechanical Properties and Structural Stability of Dental Zirconia, *Journal of Prosthodontics* 21 (5) (2012) 363–369. doi:10.1111/j.1532-849x.2011.00838.x. URL <https://dx.doi.org/10.1111/j.1532-849x.2011.00838.x>
- [10] [link]. URL <https://www.dentaldirekt.de/en/products/materials/zirconium-dioxide>
- [11] M. Inokoshi, F. Zhang, J. D. Munck, S. Minakuchi, I. Naert, J. Vleugels, B. V. Meerbeek, K. Vanmeensel, Influence of sintering conditions on low-temperature degradation of dental zirconia, *Dental Materials* 30 (6) (2014) 669–678. doi:10.1016/j.dental.2014.03.005. URL <https://dx.doi.org/10.1016/j.dental.2014.03.005>
- [12] I. Horcas, R. Fernández, J. M. Gómez-Rodríguez, J. Colchero, J. Gómez-Herrero, A. M. Baro, WSXM: A software for scanning probe microscopy and a tool for nanotechnology, *Review of Scientific Instruments*

- 78 (1) (2007) 013705–013705. doi:10.1063/1.2432410. URL <https://dx.doi.org/10.1063/1.2432410>
- [13] L. Giacomelli, G. Derchi, A. Frustaci, O. Bruno, U. Covani, A. Barone, D. D. Santis, F. Chiappelli, Surface Roughness of Commercial Composites after Different Polishing Protocols: An Analysis with Atomic Force Microscopy, *The Open Dentistry Journal* 4 (1) (2010) 191–194. doi:10.2174/1874210601004010191. URL <https://dx.doi.org/10.2174/1874210601004010191>
- [14] A. Kakaboura, M. Fragouli, C. Rahiotis, N. Silikas, Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and gloss-meter, *Journal of Materials Science: Materials in Medicine* 18 (1) (2007) 155–163. doi:10.1007/s10856-006-0675-8. URL <https://dx.doi.org/10.1007/s10856-006-0675-8>
- [15] Y. Zhang, J. Chen, L. Hu, W. Liu, Pressureless-sintering behavior of nanocrystalline ZrO₂-Y₂O₃-Al₂O₃ system, *J. Mater. Lett* 60 (2006) 2302–2305.
- [16] E. A. McLaren, N. Lawson, J. Choi, J. Kang, C. Trujillo, New high-translucent cubic-phase-containing zirconia: clinical and laboratory considerations and the effect of air abrasion on strength, *J. Compend. Cont. Educat. Dentistry* 38 (2017) 13–19.
- [17] A. Elsayed, G. Meyer, S. Wille, M. Kern, Influence of the yttrium content on the fracture strength of monolithic zirconia crowns after artificial aging, *J. Quintessence Int* 50 (2019) 344–348.
- [18] Y. J. Zhang, Making yttria-stabilized tetragonal zirconia translucent, *J. Dent. Mater* 30 (2014) 1195–1203.
- [19] (2008).
- [20] E. M. Muñoz, D. Longhini, S. G. Antonio, G. L. Adabo, The effects of mechanical and hydrothermal aging on microstructure and biaxial flexural strength of an anterior and a posterior monolithic zirconia, *Journal of Dentistry* 63 (2017) 94–102. doi:10.1016/j.jdent.2017.05.021. URL <https://dx.doi.org/10.1016/j.jdent.2017.05.021>
- [21] M. Cattani-Lorente, S. Durual, M. Amez-Droz, H. A. Wiskott, S. S. Scherrer, Hydrothermal degradation of a 3Y-TZP translucent dental ceramic: A comparison of numerical predictions with experimental data after 2 years of aging, *Dental Materials* 32 (3) (2016) 394–402. doi:10.1016/j.dental.2015.12.015. URL <https://dx.doi.org/10.1016/j.dental.2015.12.015>
- [22] V. Sergo, Room-Temperature Aging of Laminate Composites of Alumina/3-mol%-Yttria-Stabilized Tetragonal Zirconia Polycrystals, *Journal of the American Ceramic Society* 87 (2) (2004) 247–253. doi:10.1111/j.1551-2916.2004.00247.x. URL <https://dx.doi.org/10.1111/j.1551-2916.2004.00247.x>
- [23] J. Chevalier, B. Cales, D. Jmjjotacs (1999).
- [24] J. Chevalier, L. Gremillard, S. Deville, Low-Temperature Degradation of Zirconia and Implications for Biomedical Implants, *Annual Review of Materials Research* 37 (1) (2007) 1–32. doi:10.1146/annurev.matsci.37.052506.084250. URL <https://dx.doi.org/10.1146/annurev.matsci.37.052506.084250>
- [25] H. Schubert, F. Frey, Stability of Y-TZP during hydrothermal treatment: neutron experiments and stability considerations, *Journal of the European Ceramic Society* 25 (9) (2005) 1597–1602. doi:10.1016/j.jeurceramsoc.2004.03.025. URL <https://dx.doi.org/10.1016/j.jeurceramsoc.2004.03.025>
- [26] S. Deville, J. Chevalier, G. Fantozzi, J. F. Bartolomé, J. Requena, J. S. Moya, R. Torrecillas, L. A. Díaz, Low-temperature ageing of zirconia-toughened alumina ceramics and its implication in biomedical implants (2003). doi:10.1016/s0955-2219(03)00313-3. URL [https://dx.doi.org/10.1016/s0955-2219\(03\)00313-3](https://dx.doi.org/10.1016/s0955-2219(03)00313-3)
- [27] S. SCHMAUDER, H. SCHUBERT, Significance of Internal Stresses for the Martensitic Transformation in Yttria-Stabilized Tetragonal Zirconia Polycrystals During Degradation, *Journal of the American Ceramic Society* 69 (7) (1986) 534–540. doi:10.1111/j.1151-2916.1986.tb04789.x. URL <https://dx.doi.org/10.1111/j.1151-2916.1986.tb04789.x>
- [28] J. A. Muñoz-Tabares, E. Jiménez-Piqué, M. Anglada, Subsurface evaluation of hydrothermal degradation of zirconia, *Acta Materialia* 59 (2) (2011) 473–484. doi:10.1016/j.actamat.2010.09.047. URL <https://dx.doi.org/10.1016/j.actamat.2010.09.047>
- [29] T. J. Lucas, N. C. Lawson, G. M. Janowski, J. O. Burgess, Effect of grain size on the monoclinic transformation, hardness, roughness, and modulus of aged partially stabilized zirconia, *Dental Materials* 31 (12) (2015) 1487–1492. doi:10.1016/j.dental.2015.09.014. URL <https://dx.doi.org/10.1016/j.dental.2015.09.014>
- [30] T. SATO, M. SHIMADA, Transformation of Yttria-Doped Tetragonal ZrO₂ Polycrystals by Annealing in Water, *Journal of the American Ceramic Society* 68 (6) (1985) 356–356. doi:10.1111/j.1151-2916.1985.tb15239.x. URL <https://dx.doi.org/10.1111/j.1151-2916.1985.tb15239.x>
- [31] M. Yoshimura, T. Noma, K. Kawabata, S. Sōmiya (1989).
- [32] B. I. Ardlin, Transformation-toughened zirconia for dental inlays, crowns and bridges: chemical stability and effect of low-temperature aging on flexural strength and surface structure, *Dental*

- Materials 18 (8) (2002) 590–595. doi:10.1016/s0109-5641(01)00095-1.
URL [https://dx.doi.org/10.1016/s0109-5641\(01\)00095-1](https://dx.doi.org/10.1016/s0109-5641(01)00095-1)
- [33] S. Ban, H. Sato, Y. Suehiro, H. Nakanishi, M. Nawa, Biaxial flexure strength and low temperature degradation of Ce-TZP/Al₂O₃nanocomposite and Y-TZP as dental restoratives, *Journal of Biomedical Materials Research Part B: Applied Biomaterials* 87B (2) (2008) 492–498. doi:10.1002/jbm.b.31131.
URL <https://dx.doi.org/10.1002/jbm.b.31131>
- [34] S. Chowdhury, Y. K. Vohra, J. E. Lemons, M. Ueno, J. Ikeda, Accelerating aging of zirconia femoral head implants: Change of surface structure and mechanical properties, *Journal of Biomedical Materials Research Part B: Applied Biomaterials* 81B (2) (2007) 486–492. doi:10.1002/jbm.b.30688.
URL <https://dx.doi.org/10.1002/jbm.b.30688>
- [35] P. H. C. O. Prado, J. B. Monteiro, T. M. B. Campos, G. P. Thim, R. M. de Melo, Degradation kinetics of high-translucency dental zirconias: Mechanical properties and in-depth analysis of phase transformation, *Journal of the Mechanical Behavior of Biomedical Materials* 102 (2020) 103482–103482. doi:10.1016/j.jmbbm.2019.103482.
URL <https://dx.doi.org/10.1016/j.jmbbm.2019.103482>
- [36] X. Guo, T. Schober, Water Incorporation in Tetragonal Zirconia, *Journal of the American Ceramic Society* 87 (4) (2004) 746–748. doi:10.1111/j.1551-2916.2004.00746.x.
URL <https://dx.doi.org/10.1111/j.1551-2916.2004.00746.x>
- [37] F. Zhang, M. Inokoshi, M. Batuk, J. Hadermann, I. Naert, B. V. Meerbeek, J. Vleugels, Strength, toughness and aging stability of highly-translucent Y-TZP ceramics for dental restorations, *Dental Materials* 32 (12) (2016) e327–e337. doi:10.1016/j.dental.2016.09.025.
URL <https://dx.doi.org/10.1016/j.dental.2016.09.025>
- [38] M. Monzavi, F. Zhang, S. Meille, T. Douillard, J. Adrien, S. Noumbissi, H. Nowzari, J. Chevalier, Influence of artificial aging on mechanical properties of commercially and non-commercially available zirconia dental implants, *Journal of the Mechanical Behavior of Biomedical Materials* 101 (2020) 103423–103423. doi:10.1016/j.jmbbm.2019.103423.
URL <https://dx.doi.org/10.1016/j.jmbbm.2019.103423>
- [39] A. M. Alhenaki (2015).
- [40] S. A. Catledge, M. Cook, Y. K. Vohra, E. M. Santos, M. D. McClenny, K. D. Moore, Surface crystalline phases and nanoindentation hardness of explanted zirconia femoral heads, *J. Mater. Sci. Mater. Med* 14 (2003) 863–867.
- [41] G. Mitov, Y. Anastassova-Yoshida, F. P. Nothdurft, C. von See, P. Pospiech, Influence of the preparation design and artificial aging on the fracture resistance of monolithic zirconia crowns, *The Journal of Advanced Prosthodontics* 8 (1) (2016) 30–30. doi:10.4047/jap.2016.8.1.30.
URL <https://dx.doi.org/10.4047/jap.2016.8.1.30>
- [42] A. Casucci, C. Mazzitelli, F. Monticelli, M. Toledano, R. Osorio, E. Osorio, F. Papacchini, M. Ferrari, Morphological analysis of three zirconium oxide ceramics: Effect of surface treatments, *Dental Materials* 26 (8) (2010) 751–760. doi:10.1016/j.dental.2010.03.020.
URL <https://dx.doi.org/10.1016/j.dental.2010.03.020>
- [43] K. Nakamura, A. Harada, M. Ono, H. Shibasaki, T. Kanno, Y. Niwano, E. Adolfsson, P. Milleding, U. Örtengren, Effect of low-temperature degradation on the mechanical and microstructural properties of tooth-colored 3Y-TZP ceramics, *Journal of the Mechanical Behavior of Biomedical Materials* 53 (2016) 301–311. doi:10.1016/j.jmbbm.2015.08.031.
URL <https://dx.doi.org/10.1016/j.jmbbm.2015.08.031>
- [44] E. S. Elshazly, S. M. El-Hout, M. E.-S. Ali, Ytria Tetragonal Zirconia Biomaterials: Kinetic Investigation, *Journal of Materials Science & Technology* 27 (4) (2011) 332–337. doi:10.1016/s1005-0302(11)60070-4.
URL [https://dx.doi.org/10.1016/s1005-0302\(11\)60070-4](https://dx.doi.org/10.1016/s1005-0302(11)60070-4)
- [45] M. Cattani-Lorente, S. S. Scherrer, P. Ammann, M. Jobin, H. A. Wiskott, Low temperature degradation of a Y-TZP dental ceramic, *Acta Biomaterialia* 7 (2) (2011) 858–865. doi:10.1016/j.actbio.2010.09.020.
URL <https://dx.doi.org/10.1016/j.actbio.2010.09.020>
- [46] J. Shen, H. Xie, X. Wu, J. Yang, M. Liao, C. Chen, Evaluation of the effect of low-temperature degradation on the translucency and mechanical properties of ultra-transparent 5Y-TZP ceramics, *Ceramics International* 46 (1) (2020) 553–559. doi:10.1016/j.ceramint.2019.09.002.
URL <https://dx.doi.org/10.1016/j.ceramint.2019.09.002>
- [47] S. BAN, Chemical durability of high translucent dental zirconia, *Dental Materials Journal* 39 (1) (2020) 12–23. doi:10.4012/dmj.2019-109.
URL <https://dx.doi.org/10.4012/dmj.2019-109>
- [48] N. M. Moqbel, M. Al-Akhali, S. Wille, M. Kern, Influence of Aging on Biaxial Flexural Strength and Hardness of Translucent 3Y-TZP, *Materials* 13 (1) (2019) 27–27. doi:10.3390/ma13010027.
URL <https://dx.doi.org/10.3390/ma13010027>
- [49] L. Gremillard, L. Martin, L. Zych, E. Crosnier, J. Chevalier, A. Charbouillot, P. Sainsot, J. Es-

- pinouse, J. L. Aurelle, Combining ageing and wear to assess the durability of zirconia-based ceramic heads for total hip arthroplasty, *Acta Biomaterialia* 9 (7) (2013) 7545–7555. doi:10.1016/j.actbio.2013.03.030. URL <https://dx.doi.org/10.1016/j.actbio.2013.03.030>
- [50] L. Borchers, M. Stiesch, F.-W. Bach, J.-C. Buhl, C. Hübsch, T. Kellner, P. Kohorst, M. Jendras, Influence of hydrothermal and mechanical conditions on the strength of zirconia, *Acta Biomaterialia* 6 (12) (2010) 4547–4552. doi:10.1016/j.actbio.2010.07.025. URL <https://dx.doi.org/10.1016/j.actbio.2010.07.025>
- [51] M. Amaral, I. S. S. L. Weitzel, T. Silvestri, L. F. Guilardi, G. K. R. Pereira, L. F. Valandro, Effect of grinding and aging on subcritical crack growth of a Y-TZP ceramic, *Brazilian Oral Research* 32 (0) (2018) 0–32. doi:10.1590/1807-3107bor-2018.vol32.0032. URL <https://dx.doi.org/10.1590/1807-3107bor-2018.vol32.0032>
- [52] J. M. Innes (2016).
- [53] D. Bartolo, G. Cassar, N. A.-H. Husain, M. Özcan, J. Camilleri, Effect of polishing procedures and hydrothermal aging on wear characteristics and phase transformation of zirconium dioxide, *The Journal of Prosthetic Dentistry* 117 (4) (2017) 545–551. doi:10.1016/j.prosdent.2016.09.004. URL <https://dx.doi.org/10.1016/j.prosdent.2016.09.004>
- [54] A. M. O. D. Piva, L. P. C. Contreras, F. C. Ribeiro, L. C. Anami, S. E. A. Camargo, A. O. C. Jorge, M. A. Bottino, Monolithic Ceramics: Effect of Finishing Techniques on Surface Properties, Bacterial Adhesion and Cell Viability, *Operative Dentistry* 43 (3) (2018) 315–325. doi:10.2341/17-011-1. URL <https://dx.doi.org/10.2341/17-011-1>
- [55] E. A. M. Kidd, O. Fejerskov, What Constitutes Dental Caries? Histopathology of Carious Enamel and Dentin Related to the Action of Cariogenic Biofilms, *Journal of Dental Research* 83 (1_suppl) (2004) 35–38. doi:10.1177/154405910408301s07. URL <https://dx.doi.org/10.1177/154405910408301s07>
- [56] A. M. dal Piva, J. P. Tribst, L. D. Gondim, I. L. Ribeiro, F. Campos, A. Arata, R. O. Souza, Y-TZP surface behavior under two different milling systems and three different accelerated aging protocols, *Minerva Stomatologica* 67 (6) (2018) 237–245. doi:10.23736/s0026-4970.18.04138-9. URL <https://dx.doi.org/10.23736/s0026-4970.18.04138-9>
- [57] F. Zhang, K. Vanmeensel, M. Batuk, J. Hadermann, M. Inokoshi, B. V. Meerbeek, I. Naert, J. Vleugels, Highly-translucent, strong and aging-resistant 3Y-TZP ceramics for dental restoration by grain boundary segregation, *Acta Biomaterialia* 16 (2015) 215–222. doi:10.1016/j.actbio.2015.01.037. URL <https://dx.doi.org/10.1016/j.actbio.2015.01.037>
- [58] M. Matei, E.-A. Voinea, R. Rîcă, H. Manolea, L. Mogoantă, A. Salan, A. Rîcă, V. C. Dinescu, N. Cioateră, New zirconia-based materials for dental applications. Structural, morphological and histological evaluation, *Ceramics International* 45 (12) (2019) 14859–14866. doi:10.1016/j.ceramint.2019.04.217. URL <https://dx.doi.org/10.1016/j.ceramint.2019.04.217>
- [59] W. Z. Zhu, Effect of cubic phase on the kinetics of the isothermal tetragonal to monoclinic transformation in ZrO₂ (3mol% Y₂O₃) ceramics, *J. Ceram. Int* 24 (1998) 35–43.
- [60] J. Chevalier, S. Deville, E. Münch, R. Jullian, F. Lair, Critical effect of cubic phase on aging in 3 mol% yttria-stabilized zirconia ceramics for hip replacement prosthesis, *J. Biomater* 25 (2004) 5539–5545.
- [61] L. F. GUILARDI, G. K. R. PEREIRA, V. F. WANDSCHER, M. P. RIPPE, L. F. VALANDRO, Mechanical behavior of yttria-stabilized tetragonal zirconia polycrystal: Effects of different aging regimens, *Brazilian Oral Research* 31 (0) (2017) 94–104. doi:10.1590/1807-3107bor-2017.vol31.0094. URL <https://dx.doi.org/10.1590/1807-3107bor-2017.vol31.0094>
- [62] G. M. D. Souza, A. Zyklus, R. R. Ghahnavyeh, S. K. Lawrence, D. F. Bahr, Effect of accelerated aging on dental zirconia-based materials, *Journal of the Mechanical Behavior of Biomedical Materials* 65 (2017) 256–263. doi:10.1016/j.jmbbm.2016.08.023. URL <https://dx.doi.org/10.1016/j.jmbbm.2016.08.023>
- [63] G. K. R. Pereira, M. Amaral, P. F. Cesar, M. C. Bottino, C. J. Kleverlaan, L. F. Valandro, Effect of low-temperature aging on the mechanical behavior of ground Y-TZP, *Journal of the Mechanical Behavior of Biomedical Materials* 45 (2015) 183–192. doi:10.1016/j.jmbbm.2014.12.009. URL <https://dx.doi.org/10.1016/j.jmbbm.2014.12.009>
- [64] L. M. Fais, R. B. Fernandes-Filho, M. A. P. da Silva, L. G. Vaz, G. L. Adabo, Titanium surface topography after brushing with fluoride and fluoride-free toothpaste simulating 10 years of use, *Journal of Dentistry* 40 (4) (2012) 265–275. doi:10.1016/j.jdent.2012.01.001. URL <https://dx.doi.org/10.1016/j.jdent.2012.01.001>
- [65] R. Scotti, K. Z. Kantorski, C. Monaco, L. F. Valandro, L. Ciocca, M. A. Bottino, SEM evaluation of in situ early bacterial colonization on a Y-TZP ceramic: a pilot study, *J. Int. Prothodont* 20 (2007) 419–422.
- [66] M. Aboushahba, H. Katamish, M. Elagroudy, Evaluation of hardness and wear of surface treated zirconia on enamel wear. An in-vitro

study, Future Dental Journal 4 (1) (2018) 76-83.
doi:10.1016/j.fdj.2017.10.001.
URL <https://dx.doi.org/10.1016/j.fdj.2017.10.001>