

## SEM ANALYSIS OF ZIRCONIA-DENTAL MATERIAL

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### Abstract:

In dentistry, zirconia is used as a framework for dental prostheses and has been traditionally covered with porcelain to provide excellent aesthetics. The purpose of this study is to evaluate the effects of surface polishing on the properties of zirconium dioxide (Zirconia), the Tizian material, from Schutz Dental. The microstructural tests were carried out using an electron scanning electron microscope, VegaTescan. The images show a more pronounced action of the felt and Al<sub>2</sub>O<sub>3</sub> powder on the material compared to mechanical polishing by sandpapers. From the analysis of the distribution of the chemical elements we can see a good homogeneity, which leads to the maintenance of the chemical, physical and mechanical properties in all the areas of this material.

Keywords: roughness, zirconia, Al<sub>2</sub>O<sub>3</sub> powder

### INTRODUCTION

The popularity and clinical use of dental zirconia has increased due to its superior physical properties, high aesthetic potential, biocompatibility and chemical stability. In dentistry, zirconia is primarily used as framework for fixed dental prostheses and has been traditionally covered with porcelain to provide good aesthetics. [1]

Zirconia is a polycrystalline ceramic that can be found in three crystallographic forms depending on temperature: monoclinic, tetragonal and cubic [2]. Dental zirconia is stabilized by metal oxides, such as yttria (Y<sub>2</sub>O<sub>3</sub>) to allow tetragon formation at room temperature [3]. Requests may cause microfissures that generate stretching tensions and thus induce the transition from the tetragonal phase to the monoclinic phase, producing an increase in localized volume [4].

Several studies have shown that 25 µm diamond paste polishing increases zirconia resistance, while rough grinding will decrease its strength. However, some studies show that finishing the surface with fine or coarse diamond stones decreases the strength of zirconium [5, 6]. The adverse effects of

surface grinding are assumed to be caused by the heat produced by friction, [7] which exceeds the inverse transformation of temperature, causing a phase change from monoclinic to tetragonal, which may weaken zirconia [8]. In addition, increased surface roughness have a negative impact on the biological integration with development of bacterial biofilms [9-12].

### AIM OF THE STUDY

The purpose of this study is to evaluate the effects of surface polishing on the properties of dental zirconia.

### MATERIALS AND METHODS

The most used method in zirconium structure manufacturing is the CAD / CAM milling from a solid block. Zirconium blocks are obtained of dried zirconium powder, isostatically pressed and incompletely sintered. After milling, zirconium is completely sintered in an oven at 1350-1500<sup>0</sup>C to obtain the final shape, strength and physical properties.

Three samples of 0.5 cm diameter were cut from a fixed prosthesis framework (Tizian

material, from Schutz Dental) and embedded in polymeric resin. These samples were subject to the mechanical polishing with

sandpapers 600,1200 grit and felt with 0.3 µm alumina powder.

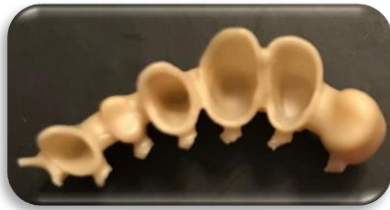
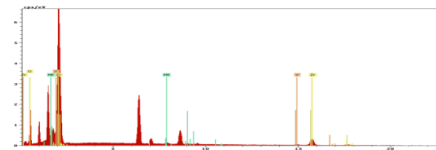


Figure 1 The main Zirconia sample

The quantitative presence of the chemical elements in the material is shown in Table 1. The chemical composition is given in mass and atomic percentages. Also, Table 1 presents the error of the EDS detector specific to each identified chemical element.

Table 1. Chemical composition of dental Zirconia



Element	YEAR	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	Error %
Zirconium	40	6632	48.16	48.16	14.95	0.43
Oxygen	8	11164	47.41	47.41	83.89	1.52
Yttria	39	761	2.88	2.88	0.92	0.15
Hafnium	72	1704	1.55	1.55	0.25	0.08
		Sum:	99.99	100	100	

The presence of the elements Y and Hf is due to the zirconium and they do not negatively influence the corrosion resistance or the hardness of the dental material.

The microstructural tests were carried out using an electron scanning electron microscope (SEM) by VegaTescan, the LMH II model (fig. 2).



Figure 2. Electron scanning microscope

## RESULTS AND DISCUSSIONS

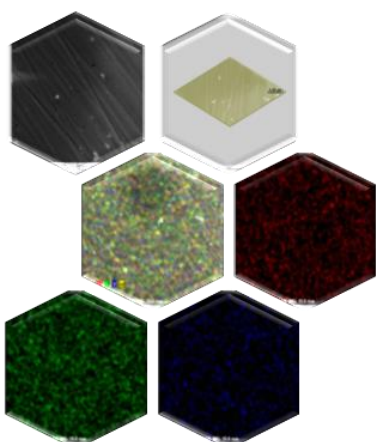
The experimental results obtained by scanning electron microscopy are presented for the analysis of the effects of surface polishing techniques.

The 3D) images of the dental zirconia surface condition after mechanical polishing with sandpapers 600, 1200 grit and felt with 0.3 µm alumina solution.

The images (micrometric and submicrometric scale) show the state of the surface without ultrasonic cleaning or methods for removing scrap materials used for grinding.

The images in Figure 3 show a surface with a uniform orientation of the surface asperities, located in the dimensions of 2-3  $\mu\text{m}$  between them and with submicronic heights (300-500 nm, Fig. 3 c) and d)).

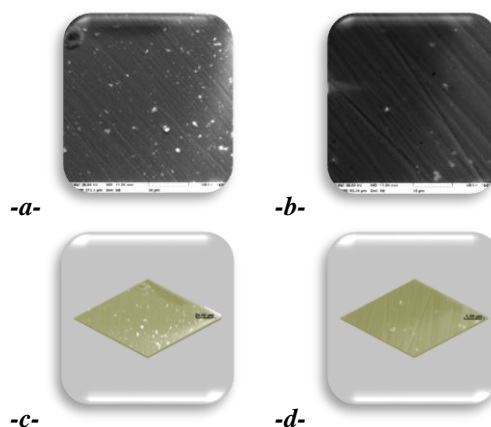
Figure 4 shows the distribution of the Zr, O, Y and Hf elements of which Y and Hf are accompanying elements on the surface of a zirconia dental element.



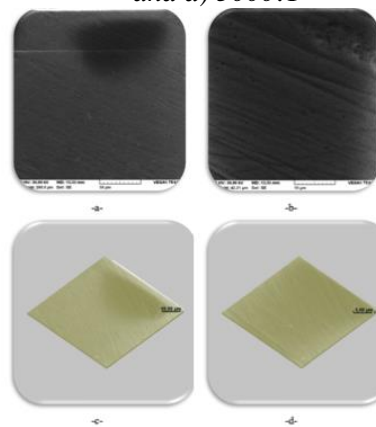
**Figure 4.** The distribution of Zr, O, Y and Hf elements

From the analysis of the distribution of the component elements we can see a good homogeneity of the chemical elements in the alloy, which leads to the maintenance of the chemical, physical and mechanical properties in all the areas of this material.

Figure 5 shows the surface of dental zirconia, 2D (a and b) and 3D (c and d) images polished to sandpaper 1200 grit a) and c) 1000:1 respectively b) and d) 5000:1. A lower surface roughness is observed compared to the 600 grit polished sample. A surface structure was made at a micron level.



**Figure 3.** Zirconia dental material, 2D (a and b) and 3D (c and d) images polished to 600 grit a) and c) 1000:1 respectively b) and d) 5000:1

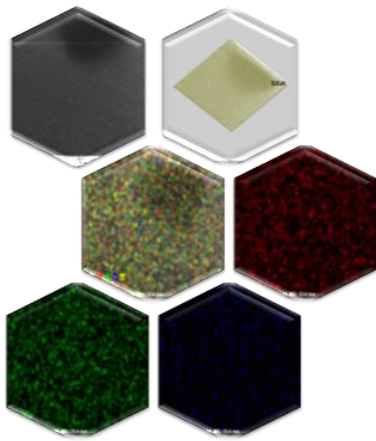


**Figure 5.** Dental Zirconia, 2D (a and b) and 3D (c and d) images polished to 1200 grit a) and c) 1000:1 respectively b) and d) 5000:1

The distances between the processed material areas are at submicron level (300-500 nm) and the surface roughness is below 300 nm. The surface of the material behaves similarly to the previous process.

The 3D appearance of the surface shows the homogeneity of the material.

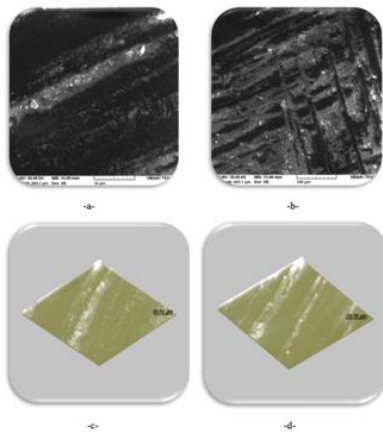
The distribution of the main chemical elements is presented in fig.6.



**Figure 6.** The distribution of Zr, O, Y and Hf elements

From the distribution of the main components (Zr and O) and accompanying elements (Y and Hf) shown in fig.6, the chemical homogeneity of the material is observed.

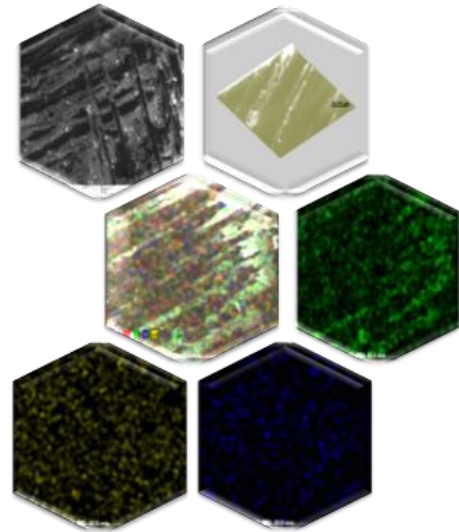
The images from figure 7, show a more pronounced action of the felt with alumina powder on the material compared to mechanical polishing by sandpapers.



**Figure 7.** Dental Zirconia, 2D (a and b) and 3D (c and d) images polished to felt a) and c) 1000:1 respectively b) and d) 5000:1

The general distribution of the elements (fig.8) indicates an accentuated oxidation of

the areas from which the material was detached.



**Figure 8.** The distribution of Zr, O, Y and Hf elements

## CONCLUSION

It has been shown that surface grinding significantly affects the state of the surface of the zirconium samples.

The images show a more pronounced action of the felt + Al powder $2O_3$  on the material compared to mechanical machining by grinding. It has been noticed that mechanical polishing to the sandpaper 1200 grit exhibits the smooth surface.

The chemical analysis of the material identified elements of oxygen (to one energy) and zirconium (at three energies) as well as the accompanying elements yttria and hafnium.

From the analysis of the distribution of the component elements we can see a good homogeneity of the chemical elements in the alloy, which leads to the maintenance of the chemical, physical and mechanical properties in all the areas of this material.

## BIBLIOGRAPHY

1. C.M.B. Hoa, H. Dingb, X. Chenc, J.K.H. Tsoib, M.G. Botelho, The effects of dry and wet grinding on the strength of dental zirconia, *Ceramics International*, Volume 44, Issue 9, p.10451-10462, 2018.

2. JR Kelly, I. Denry, Stabilized zirconia as a structural ceramic: an overview, *Dent. Mater.* 24 (2008) 289-298  
Malkondu Ö, Tinastepe N, Akan E, Kazazoğlu E. An overview of monolithic zirconia I in dentistry. *Biotechnol Biotechnol Equip*, 30 (4), p.644-52, 2016.
3. JB Quinn, V. Sundar, IK Lloyd, Influence of microstructure and chemistry on fracture toughness of dental ceramics, *Dent. Mater.*, 19, p.603-611, 2003.
4. AG Evans, Perspective on the development of high-toughness ceramics, *J. Am. Ceram. Soc.* 73, p.187-206, 1990.
5. Anselmi-Tamburini U, Woolman JN, Munir ZA. Transparent nanometric cubic and tetragonal zirconia obtained by high-pressure pulsed electric current sintering. *Adv Funct Mater*, 17 (16), p.3267-73, 2007.
6. D. Chaysuwan, K. Sirinukunwattana, K. Kanchanatawewat, G. Heness, K. Yamashita, Machinable glass-ceramics forming as a restorative dental material, *Dent. Mater. J.* 30, p.358-367, 2011.
7. Heintze SD, Rousson V. Survival of zirconia- and metal-supported fixed dental prostheses: a systematic review. *Int J Prosthodont*, 23, p.493-502, 2010.
8. Ioannidis A, Bindl A. Clinical prospective evaluation of zirconia-based three-unit posterior fixed dental prostheses: up to ten-year results. *J Dent*, 47, p.80-5, 2016.
9. S. Abbas, S. Maleksaeedi, E. Kolos, A. Ruys, Processing and properties of zirconium-gouged alumina prepared by gelcasting, *Materials*, 8, p.4344-4362, 2015.
10. A.A. Ciocan-Pendefunda, N.C. Forna, Starea de suprafață a restaurărilor fixe și influența asupra parodontiului, *The Medical-Surgical Journal*, 116 (1), p.317-323, 2012.
11. ER Baci, D Chicet, M Mareș, C Munteanu, C Baci, NC Forna, Influence of finishing techniques of non-noble alloys used in prosthetic restoration on *Candida albicans* biofilm development in wet systems, *Environmental Engineering and Management Journal*, Vol.11, No. 5, p.1015-1022, 2012.
12. Baci RE, Grădinaru I, Toma Ș, Baci M, Baci C, Forna CN, Predictive analysis of surface quality-*Candida albicans* biofilm relations, *Romanian Journal of Oral Rehabilitation* Vol.7, No. 3, p.47-49, 2015