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MICROSTRUCTURAL AND GRAIN SIZE ANALYSIS OF ZIRCONIUM MATERIALS FOR DENTAL PROSTHESES RESTORATION

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Abstract: Zirconia is a ceramic material with great potential in the biomedical field, it shows properties such as biocompatibility, low reactivity, and optical qualities. Its use as a dental material has been reported with excellent mechanical results, however it presents a high opacity limitation. A characterization of the microstructure was carried out with SEM on Zr specimens thermally etched at 1200°C, using a curing and cooling speed of 10°C/min. The average grain size was measured from the 10,000x micrographs using the linear intercept method in ImageTool software. The microstructure of the zirconium materials was obtained through micrography. The relationship between the modifier content and the crystallographic phase with the grain size was identified; according to the linear intersection method, there is a relationship between the modification and the reported grain size. Simplifying design, manufacturing, and aesthetics with digital developments in complex restorations, improves monolithic ceramic restorations. Increasing the amount of Y_2O_3 results in a higher cubic phase content and a larger grain size and reduces light scattering.

Keywords: zirconia, biocompatible ceramic, microstructure, grain size, dental restoration.

INTRODUCTION

Zirconia is one of the ceramics with the greatest potential to be a substitute material in biomedical applications, with biocompatibility properties, low reactivity, and excellent optical properties. For 30 years, the use of stabilized zirconia as a dental material with excellent mechanical results (hardness and resistance to fracture) has been reported, however, it presents a high opacity limitation, which is why it has been used for crown structures and fixed dental prostheses, which are covered with translucent porcelain, in a similar way to what is reported in the literature for traditional porcelain fused to metal restorations. However, in these modifications, chipping, and fracture, associated with the residual thermal stresses produced by the manufacturing process, are the main technical limitations reported (Luna-Domínguez et al., 2023).

Nowadays, the techniques used in the handling and transformation of metals are considered applicable to different ceramic systems. Currently, phase transformations, alloying, cooling, and tempering are applied to different zirconia ceramic systems. The significant increase in fracture toughness, ductility, and impact strength of the composite has narrowed the gap between the physical properties of ceramics and metals. Furthermore, recent developments in oxide-free and higher-hardness ceramics, as well as huge investments in the dental industry, have also created high expectations for the potential application of ceramic materials in clinical dentistry (Sen et al., 2017). From a technology point of view, zirconia appears to have excellent short-term performance; However, there is no data on the long-term longevity of this compound, which is why its future remains unknown.

The use of this type of materials in the dental field has been recently reported, Kolakarnprasert et al., in 2019, established that to identify the advantages of the application of multilayer zirconia, a comprehensive understanding of its chemical composition, microstructure, resistance to low-temperature degradation and translucency properties is necessary. They evaluated 3 different grades of multilayer zirconia, analyzing the materials individually and their layers; the chemical composition, zirconia phase fractions, microstructure, LTD resistance, and translucency properties were determined. They concluded that there were no important differences between the layers, but the 3 materials were very different. Translucency was similar between layers. For each grade of multilayer zirconia, the layers only differ in the types of pigments and their contents, producing natural color gradients.

Andrade-Guel et al., in 2019, reported the use of zirconium dioxide (ZrO_2) as a ceramic with good properties for applications in the medical, chemical, and pharmaceutical fields. They attribute this to its amphoteric qualities, with three crystalline phases, monoclinic, tetragonal, and cubic, which gives different properties to these materials. They analyzed the synthesis methods of ZrO_2 and its biomedical applications, evaluating the hydrothermal, precipitation, solvothermal and sol-gel methods. Concluding that the process conditions (ultrasound energy and microwave radiation) allow reducing reaction times and increasing energy efficiency. They also established that the synthesis methods modify the ZrO_2 properties, influencing the potential application of the product obtained, including bone replacements, dental prostheses, and drug release.

Marcelo et al., in 2020, published a bibliographic review with the analysis of 48 articles chosen based on the use and application criteria of the zirconium dioxide material. They found that all prosthetic restorations where zirconia dioxide was used, regardless of the way it was used, showed a very similar clinical performance over time (3 years), and that after that time, it depends on the use, composition, and choice of the case. They concluded that the clinical success of restorations where zirconium dioxide was used is due to the multiple mechanical advantages, structural modifications, added to the addition of additives for stabilization, profiling it as an ideal material for its application in both aesthetics and mechanical support.

The present work aims to analyze and evaluate the modifications to the physicochemical properties of zirconia with respect to the concentration of yttrium used, to demonstrate that the microstructural and size (grain) properties are favorable for the potential application of these materials in dental restorations and simplification of prosthetic treatment.

METHODOLOGY

MATERIALS

The multilayer zirconia systems used were those commercially available (Katana™; Kuraray Noritake). Table 1 shows the nomenclature of the three systems employed.

Id	Nomenclature	Commercial abbreviations
ZUT	Zirconia Ultra Translucent Multi Layered	UTML
ZST	Zirconia Super Translucent Multi Layered	STML
ZHT	Zirconia High Translucent Multi Layered	HTML

Table 1. Nomenclature of the multilayer zirconia systems used.

DISC SYNTHESIZING AND MANUFACTURING

Multilayer disc-shaped specimens (15 mm diameter and 1.0 mm thickness) were designed using DentalCAD 3.0 Galway software. Subsequently, 12 samples for each zirconia material (ZUT, ZST and ZHT) were milled from pre-synthesized discs (98 mm x 18 mm) using a milling machine (350i timesicore GmbH). The samples were removed from the zirconia discs with the help of a micromotor using a diamond bur and synthesized following the conditions in Table 2.

Id	Temperature increase rate	Temperature superior	Temperature decrease rate	Flexural strength
ZUT	10˚C/min	1550˚C for 2 h	10˚C/min	557 MPa
ZST	10˚C/min	1550˚C for 2 h	10˚C/min	748 MPa
ZHT	10˚C/min	1500˚C for 2 h	10˚C/min	1125 Mpa

Table 2. Sintering conditions and mechanical properties of multilayer zirconia systems.

Then, all samples were polished with rubber discs (Eve Diapol) using a low speed micromotor (Schick GmbH) with a speed of 10,000 rpm. The samples were polished in one direction for 30 seconds, rotated 90°, and polished for another 30 seconds. The polishing system consists of 2 steps using 2 different grain rubber discs. A full one-minute polishing was performed for each sample. Subsequently, the samples were cleaned in ultrasound with distilled water for 10 minutes.

MICROSTRUCTURAL AND GRAIN SIZE ANALYSIS

The characterization of the microstructure was carried out by scanning electron microscopy (SEM) on zirconia specimens previously thermally etched at 1200ºC for 1 hour, using a curing and cooling speed of 10ºC/min. The average grain size was measured on SEM micrographs at 10,000x magnification using the linear intercept method in ImageTool software.

RESULTS

SYNTHESIS

To create the test pieces, the synthesis process was carried out under the conditions described in the methodology based on the specimen designed in DentalCAD 3.0 Galway (figure 1). This casting is carried out with a high-precision laser that melts the material creating each layer until the final piece is obtained.

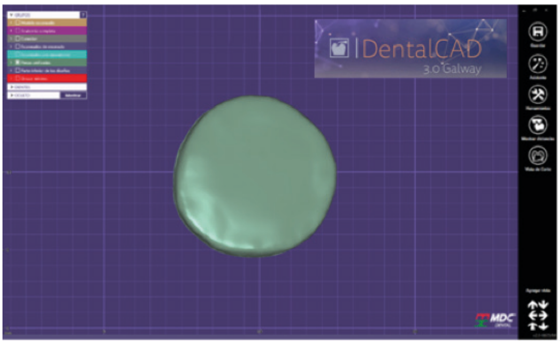

Figure 1. Disc-shaped sample designed by DentalCAD 3.0 Galway.

The layers obtained are of great complexity and detail (figure 2), the synthesis process made it possible to reproduce the most demanding angles and areas, either due to their curved morphology or due to the any special requirements in the production of the piece.

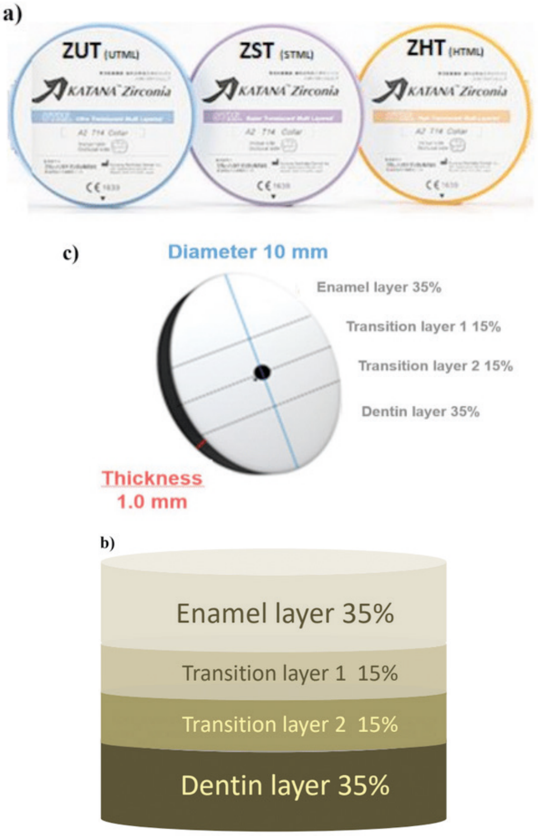

Figure 2. a) Batches of multilayer zirconia used (Katana™ Kuraray Noritake), b) Scheme of the gradient of the multilayer zirconia disc. c) Layers of a zirconia sample segment.

COMPOSITION

The yttrium oxide content was different among the zirconium materials studied. ZUT showed the highest yttrium content, 12.30 wt%, ZST had 10.18 wt%, and ZHT had the lowest content, 7.18 wt% (Figure 3).

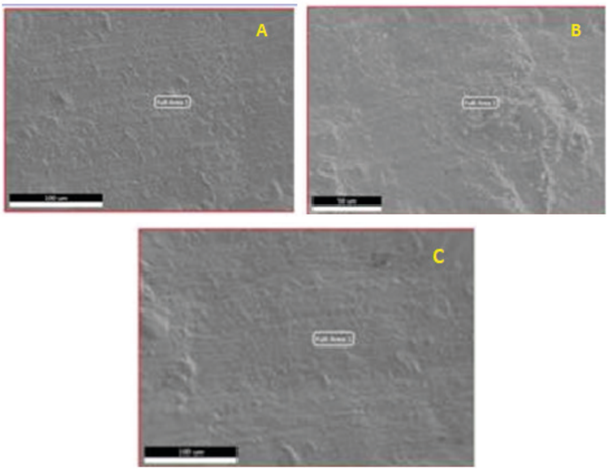

Figure 3. Structural comparison of the percentage content of yttrium (% weight) in the three batches of zirconium material a) ZUT, b) ZST and c) ZHT.

Table 3 reports the compositional percentage values, obtained by EDX, of the different batches of multilayer zirconia that were evaluated. It is observed that the yttrium content decreases with the increase of zirconium present in the sample, this provides a greater content of cubic phase in the piece, an such arrangement allows pieces with compatible mechanical and aesthetic qualities.

Batch	Compound	Weight %	Atomic %	Net. Int.	Error %
ZUT	Y_2O_3	12.30	7.11	18.66	9.43
	ZrO_2	87.70	92.89	122.44	4.62
ZST	Y_2O_3	10.18	5.82	14.58	12.37
	ZrO_2	89.82	94.18	118.90	4.45
ZHT	Y_2O_3	7.18	4.05	9.92	15.76
	ZrO_2	92.82	95.95	119.11	4.28

Table 3. Comparison of yttrium content (wt%) in multilayer zirconia materials

MICROSTRUCTURE AND GRAIN SIZE

Figure 4 shows representative micrographs of the 3 batches of zirconia studied. No porosity was observed in any of the micrographs. The microstructures agree with the yttrium and cubic phase contents, with the ZUT sample having the largest grain size and ZHT the smallest. The average grain size determined by the linear intercept method confirmed these observations, for ZUT 4.00 ± 0.85 µm, ZST 2.80 ± 0.17 µm and ZHT 0.63 ± 0.03 µm, which coincide with those reported elsewhere (Camposilvan et al., 2018; Inokoshi et al., 2018). Moreover, the high dispersion of the grain size in the ZUT sample can be observed in the micrographs, which has varied sizes that produces a large deviation in the results (standard deviation). In contrast, ZHT is more homogeneous, and a show a smaller standard deviation, which refers to a narrower grain size distribution (Kolakarnprasert et al., 2019).

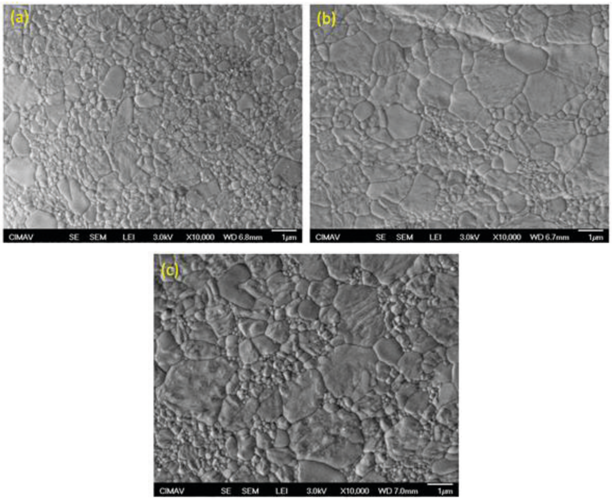

Figure 4. Representative FE-SEM micrographs of the batches of zirconium materials studied a) ZUT, b) ZST and c) ZHT.

CONCLUSIONS

The results obtained on yttrium content and microstructural characteristics were consistent with bibliographic reports and manufacturer information, showing that ZUT has the highest yttrium content while ZHT, the lowest content. Despite observing this similarity between the studies, some discrepancies were observed between the specific values of yttrium content, but this is due to the different techniques used for its measurement. Likewise, the results of the present study are consistent with other published that supports the alternative of implementing the complete multilayer zirconia system (KatanaTM, Kuraray) in total restorations performed through a digital workflow.

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