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Characterization of Ceramic Powders Used in the InCeram Systems to Fixed Dental Prosthesis

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InCeram (Vita Zahnfabrik- Germany) is known as a high strength ceramic being used for core crowns and for fixed partial denture frameworks. InCeram system consists of slip-casting technique which is used for to build the framework, which is then pre-sintered obtaining an open-pore microstructure. The material gains its strength by infiltration of the lanthanum glass into the porous microstructure. In this work, commercial alumina (Al_2O_3), alumina-zirconia (Al_2O_3-ZrO_2) and glasses lanthanum oxide-rich powders, used in InCeram system, were characterized, using x ray diffraction, dilatometry and scanning electron microscopy. The characteristics of these powders were related aiming to consider their substitution for new ceramic materials.

Keywords: *ceramics, glasses, dental prosthesis, InCeram System, characterization*

1. Introduction

The development of new technologies for the production of biomaterials has been motivated by the demand for materials, capable of bearing new specifications and applications[1,2]. The use of ceramics as biomaterials started in the 1970's, and since then, a continuous improvement of these materials, in several applications, have been noted.

Since few years ago, significant advances were obtained in the development of dental restoration techniques using implantation systems composed of ceramic materials, also called "metal free" systems. These materials present advantages due to the excellent performance of its functional properties, mainly aesthetic, biocompatibility and chemical resistance. There is a trend of substitution of the metallic restoration substructure by toughened ceramic materials, aiming the improvement of the prosthesis aesthetic.

Ceramic materials based on alumina (Al_2O_3) and zirconia (ZrO_2) are used as materials of dental infrastructure due to their excellent properties, such as strength, corrosion resistance and biocompatibility[3-5]. Usually, in these systems prosthesis-implantation, Al_2O_3, ZrO_2 and their composites, are prepared according to the geometry of the intended restoration, from glue by barbotine, which is succeeded by a pre-sintering, generating a compacted structure with resistance intermediate strength. Later, this material suffers an infiltration from lanthanium based glass, generating a secondary glassy phase that fills the interconnected pores, increasing the strength and toughness by the stress field generation around the ceramic matrix, resulted by difference of the coefficient of thermal expansion between both phases.

Pure zirconia cannot be used in the fabrication of components, without the addition of stabilizers. Yttrium-stabilized zirconia (Y-TZP) became a popular alternative to the alumina as structural bioceramic, because it is also inert in physiological media and presents greater flexural strength and fracture toughness and also low Young' modulus[5-7].

The objective of this work was the characterization of commercial InCeram dental ceramic powders, correlating its characteristics with the sintering phenomena.

2. Experimental Procedure

2.1. Starting powders

Commercial Al_2O_3 and Al_2O_3-ZrO_2 ceramic powders and their respective glasses (In-Ceram Alumina and InCeram zirconia - VITA Zahnfabrick-Germany), were used in this work. Table 1 shows the composition of the different powders.

2.2. Characterization

The coefficient of thermal expansion (CTE) of the previously consolidated bulk ceramics and glasses, was measured using an alumina rod dilatometer (BAHR Thermoanalyse GmbH 2000 Model DIL801L).

The crystalline phases of the powders were identified by x ray diffraction using Seifert - IsoDebyeflex 1001 diffractometer. The data were collected in the 2θ interval between 20° and 70°, with 0.05° steps using 2 seconds of counting time.

Morphological aspects, chemical composition and average particle sizes were obtained, using scanning electron microscopy (LEO 1450VP Scanning Electron Microscope), with coupled Energy Dispersive Spectrometer (EDS) - OXFORD INCA.

3. Results and Discussion

Figure 1 shows x ray diffractogram patterns of the commercial powders: a) In Ceram alumina, b) InCeram zirconia, c) InCeram alumina-Glass Powder, d) InCeram zirconia-Glass Powder.

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Table 1. Composition of the starting powders used in this study.

Material	InCeram alumina		InCeram zirconia	
	InCeram alumina powder (wt. (%))	Glass powder – InCeram alumina (wt. (%))	InCeram zirconia powder (wt. (%))	Glass powder – InCeram zirconia (wt. (%))
Al_2O_3	100	14-17	67	14-18
ZrO_2	-	-	33	-
SiO_2	-	14-17	-	14-18
B_2O_3	-	12-15	-	11-15
TiO_2	-	3-5	-	2-7
La_2O_3	-	39-48	-	25-30
CeO_2	-	2-5	-	6-10
CaO	-	2-4	-	4-8
ZrO_2	-	-	-	1-4
Y_2O_3	-	-	-	2-6

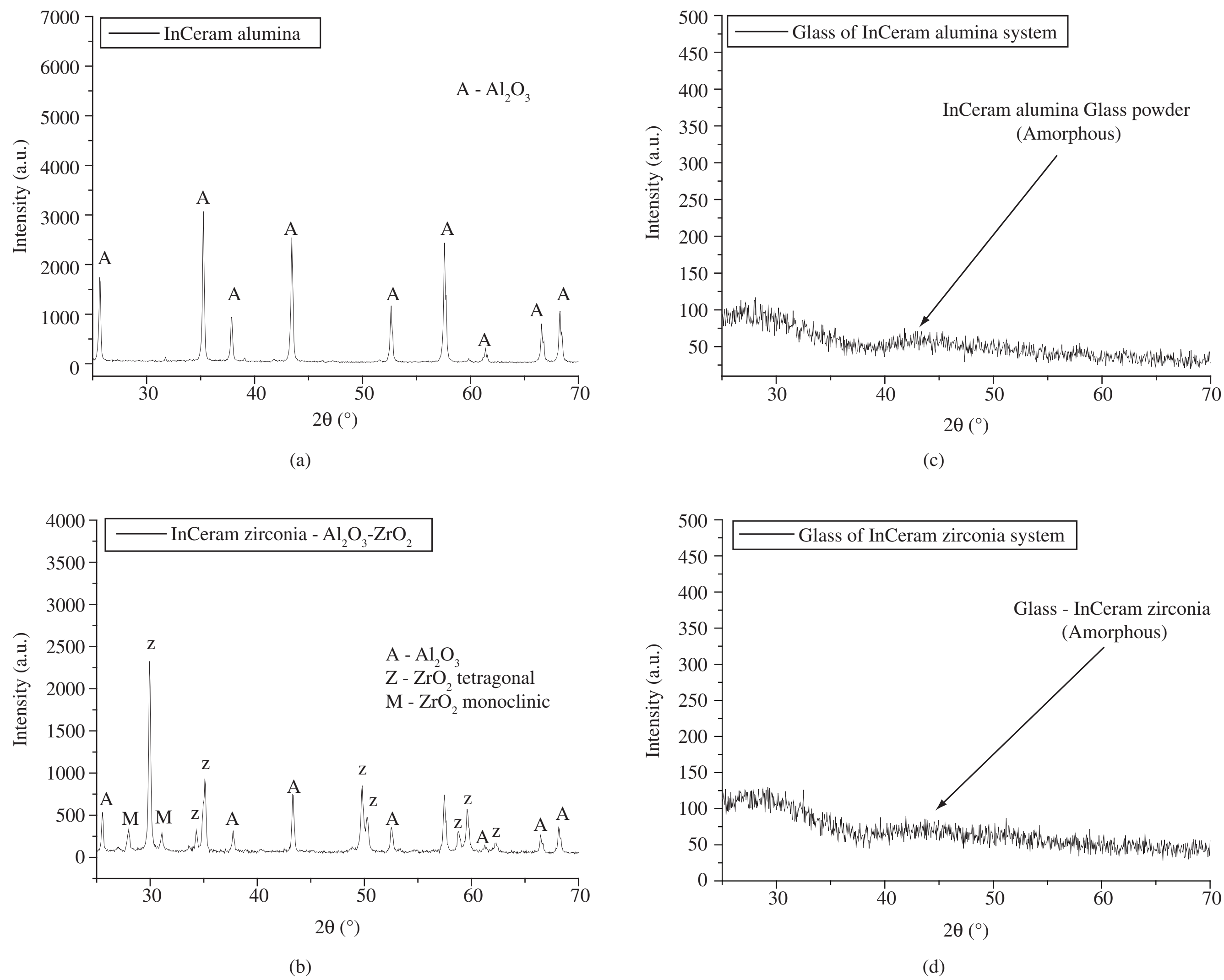


Figure 1. X ray diffractogram patterns of ceramic powders of InCeram systems.

In the Figure 1a, it can be observed that the InCeram alumina system is composed by α-Al_2O_3 crystalline phase. The x ray diffractogram pattern of InCeram zirconia powder (Figure 1b) presents crystalline phases α-Al_2O_3, tetragonal ZrO_2 and monoclinic ZrO_2.

Based on the composition presented in Table 1 and confirmed by x ray diffractogram patterns, the InCeram alumina powder presents 100% of Al_2O_3 phase, and the InCeram zirconia powders are composed of 67% of Al_2O_3 and 33% of ZrO_2, indicating that this material is a ZTA (Zirconia Toughened Alumina) which ZrO_2 particles are toughening agents[8-10]. Then, the tetragonal ZrO_2 grains, uniformly dispersed in the Al_2O_3 matrix, blocks the growth of cracks due to tetragonal-monoclinic phase transformation which promotes compressive stress around ZrO_2 grains[9]. This way, it is expected that InCeram zirconia system presents better fracture toughness that InCeram alumina system.

The x ray diffractogram patterns of the glass of both InCeram systems, Figure 1c-d presents similar characteristics, without the presence of crystalline peaks, characterizing them as glass material.

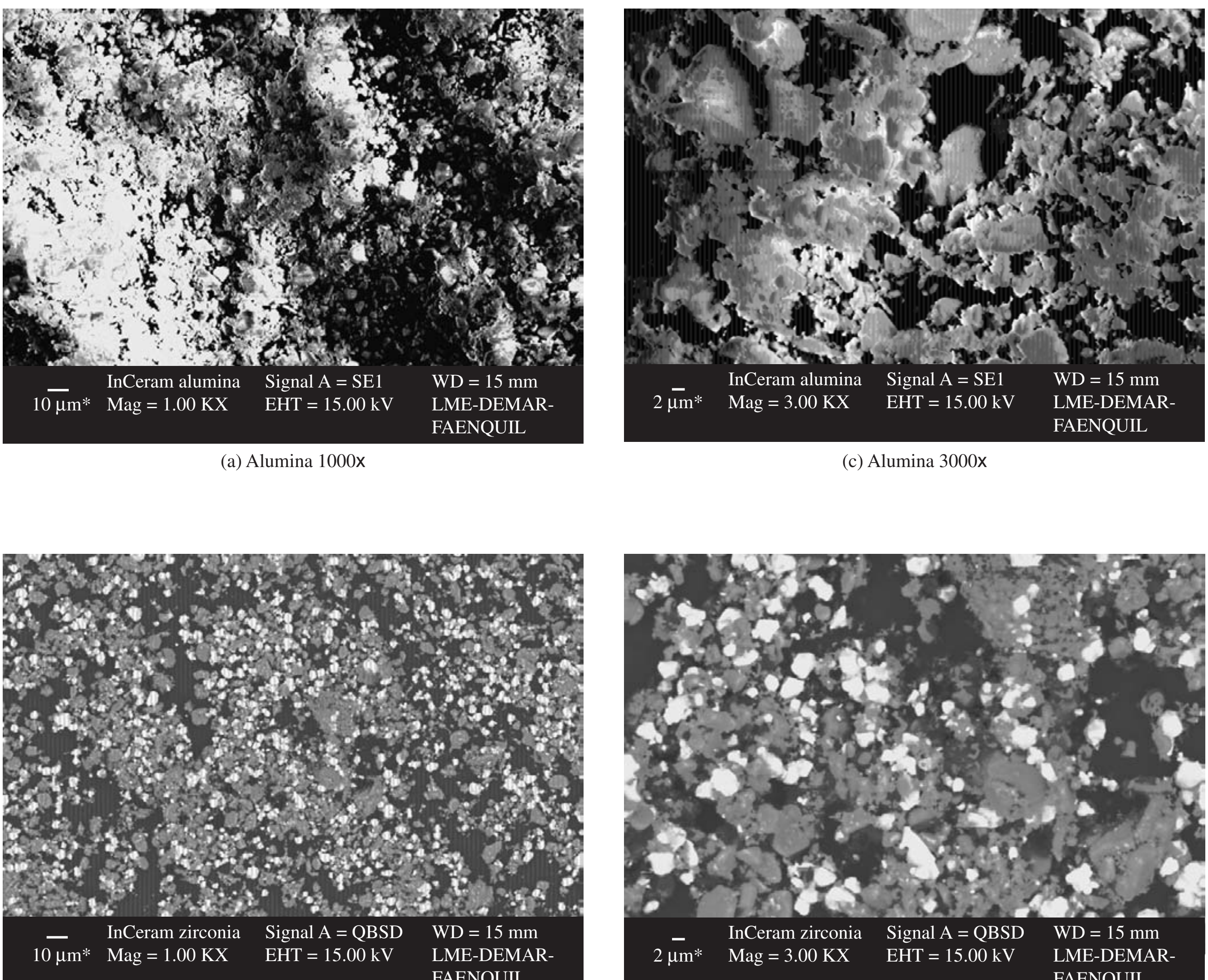
Figure 2 shows micrographs of InCeram ceramic powders.

It can be observed in the Figure 2a and 2c, only the presence of fine particles of Al_2O_3 with average grain size around 1 µm, coherent with the chemical analysis and the x ray diffraction pattern previously presented. In the Figures 2b and 2d, it can be observed fine particles of ZrO_2 (white phase) homogeneously distributed in the Al_2O_3 matrix (gray phase), both with sizes around 1 µm.

It is well known[11] that fine particles present a high specific surface area per volume unit. Moreover, an ample distribution of particles size promotes a better packing of these particles during the pressing or glue of the ceramic parts. These characteristics make possible a greater accommodation between particles of different sizes and greater contact between them, facilitating the sintering process. An efficient sintering generates materials with good mechanical resistance, desired property for dental implantation parts.

Figure 3 presents the powder morphology of the respective glasses of the InCeram systems.

Figure 3 shows that both glass powders present particles with varied sizes. It is noticed the presence of particles varying from 1 to 200 µm. This large particles size distribution difficult the easy powder infiltration by the porous pre-sintered ceramic, during the glass melting infiltration process. These characteristics of the particles must justify the high infiltration times demanded by the development of the InCeram dental crowns, (between 3 and 6 hours). Based on these results, it can be proposed a reduction of the particles sizes of the glass, facilitating the infiltration of theses glasses during melting. This way, lower infiltration times can be proposed to manufacture the infiltrated ceramic composites.



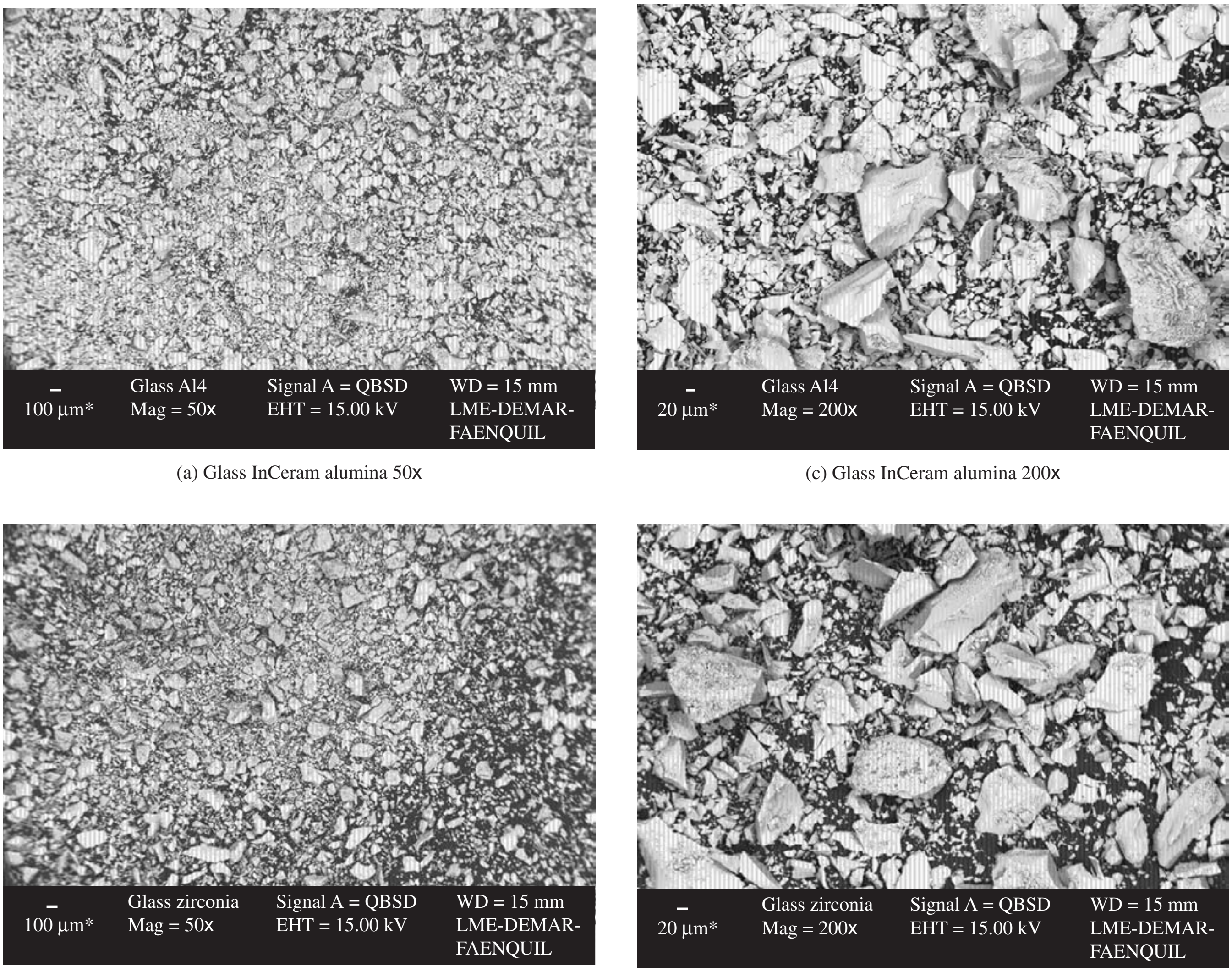
(a) Alumina 1000x

(c) Alumina 3000x

(b) Zirconia 1000x QBSD

(d) Zirconia 3000x QBSD

Figure 2. SEM micrographs of the InCeram alumina and alumina-zirconia powders (images obtained by secondary electrons or backscatering electrons emission-QBSD).



(a) Glass InCeram alumina 50x

(c) Glass InCeram alumina 200x

(b) Glass InCeram zirconia 50x

(d) Glass InCeram zirconia 200x

Figure 3. SEM micrographs of the InCeram glass powders: a,c) InCeram alumina; and b,d) InCeram zirconia.

Table 2. Coefficient of thermal expansion of the InCeram ceramic powders.

Materials	Coefficient of thermal Expansion (CTE) α (10^{-6} / K)
Al_2O_3 – InCeram alumina	7.65
Al_2O_3-ZrO_2 – InCeram zirconia	8.62
Glass InCeram alumina	7.03
Glass InCeram zirconia	8.07

Materials for infrastructure in dental implantations of InCeram system, are basically obtained by the glue technique: Al_2O_3 or Al_2O_3-ZrO_2 powders are mixed with optimized water content and deflocculant, obtaining a suspension that is shed in the plaster mold with the geometry of the intended restoration. The suspension is placed until the plaster mold absorbs enough water to obtain the desirable thickness. After this, the liquid in excess is removed and time is given, so the sample shrinkage occurs allowing its withdrawal from the mold. This sample, also called green body, is submitted to the drying for elimination of the water. The material suffers pre-sintering (lower 1150 °C) obtaining samples with low-relative density presenting open porosity and remaining itself the initial dimensions.

The glass powder is casting and infiltrated in the pre-sintered ceramic for the elimination of open-porosity. The bulk is cooled and it is observed the generation of stress fields due to the difference of the coefficient of thermal expansion between the materials, ceramic matrix and glass. These stress fields exert a contrary force to the crack growth, offering crack propagation resistance. This process results in the reduction of the sintering temperature and the increasing of fracture toughness, in relation to pure sintered alumina.

Table 2 presents results of coefficient of thermal expansion for consolidated materials of InCeram systems.

The results indicate that materials of InCeram alumina system present coefficient of thermal expansion (CTE) of 7.65 x 10^{-6}/K and 7.03 x 10^{-6}/K for Al_2O_3 and the glass used in infiltration, respectively. InCeram zirconia powders present CTE of 8.62 x 10^{-6}/K and 8.07 x 10^{-6}/K for Al_2O_3-ZrO_2 composite and glass used in the infiltration, respectively. It can be observed in both systems, a difference lower than 10% between CTE-matrix and CTE-glass. Besides, the CTE of the glass is lower than the ceramic matrix. In this case, after the infiltration of the glass in the ceramic matrix, during the cooling, this difference of CTEs, promotes a compressive stress field around grain boundary, improving the fracture toughness of the infiltrated ceramic.

4. Conclusions

In this work, the characteristics of the particle morphology and crystalline phases of the commercial InCeram ceramic powders are presented and discussed. Based on the results presented, it is concluded that these ceramics must present open porosity to allow the entrance of the glass during infiltration, eliminating the pores. It is suggested that glass with lower coefficient of thermal expansion than ceramic matrix must be required by the generation of stress field around of ceramic matrix, improving fracture toughness. So, the refinement of the glass powder allows the reduction of the sintering times.

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