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Characterization of glass-infiltrated alumina-based ceramics

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Abstract

Objective—characterize the microstructure, composition, and important properties of glass-infiltrated alumina-based ceramics similar to the In-Ceram system.

Methods—Materials used were: IA- In-Ceram Alumina (Vita); IAE- IA electrophoretically deposited (Vita); AEM- IA using a vacuum driven method (Vita); VC- Vitro-Ceram (Angelus); TC- Turkom-Cera (Turkom-Ceramic); CC- Ceramcap (Foto-Ceram); and AG- Alglass (EDG). Ceramic specimens were fabricated following manufacturers' instructions and ISO6872 standard and polished successively through 1 μ m alumina abrasive. Semi-quantitative and qualitative analyses were performed using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and stereology (Vv). The elastic modulus (E) and Poisson's ratio (ν) were determined using time-of-flight data measured in an ultrasonic pulser/receiver and the density (ρ) was determined using a helium pycnometer. Vicker's indentation was used to calculate hardness (H). Bar specimens (25 $\times$ 4 $\times$ 1.2mm³) were loaded in three-point bending to fracture using a universal testing machine with cross-head speed of 1mm/min. Flexural strength (σ_{3P}) was calculated and statistically analyzed using ANOVA, Tukey ($\alpha=0.05$) and Weibull (m = modulus, σ_0 = characteristic strength).

Results—SEM and EDS analyses revealed similar microstructure for all ceramics, except for a lead-based matrix in CC and a zirconia phase in VC. TC, AG and CC showed significantly lower mean σ_{3P} values than the other ceramics ($p < 0.05$). AEM showed the greatest m (16).

Conclusion—Despite few differences in microstructure and composition, the IA, IAE, AEM and VC ceramics have similar properties.

Significance—The glass-infiltrated alumina-based ceramics from different manufacturers presented distinct characteristics. It is necessary to characterize new commercially available materials to understand their properties.

Keywords

microstructure; composition; physical properties; ceramic

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1. Introduction

The clinical success of all-ceramic restorations and the persistent patient demand for esthetic treatment resulted in the development and introduction of many ceramic systems. This continuous dynamic evolution in ceramic materials and technology has sustained a progressive research interest in dental ceramic restorations. This constant improvement is based on the excellent properties of ceramics, mainly their biocompatibility, chemical stability, and great capacity to mimic dental tissues.

The all-ceramic concept was introduced by McLean in the 1960s [1,2] using conventional manufacturing techniques. He reported that the strength of the alumina-reinforced ceramic was dependent on the volume fraction of crystalline material incorporated in the glass matrix, but it was limited by failure to eliminate porosity during sintering [2].

The reduction of defects has been the goal of all ceramic systems to improve strength and fracture toughness. It has been reported that microstructure, composition and processing are the controlling factors in the development of desired mechanical properties [3,4]. The manufacturer of the In-Ceram system (Vita Zahnfabrik, Bad Sackingen, Germany) also pursued this goal and improved the concept with a new technology using crystalline phases, such as alumina, in a slip casting procedure for the framework. The In-Ceram Alumina, introduced in 1989, was the first all-ceramic restorative system used for 3-unit anterior fixed partial dentures (FPDs). Through this technique, the technician applies and sinters a slurry of densely packed (70–80 wt%) alumina to a refractory cast at 1120°C for 10 h. This produces a porous structure of alumina particles, which is infiltrated by capillary action with lanthanum oxide-based glass in a second firing at 1100°C for 4 h to eliminate porosity, increase strength, and limit potential sites for crack propagation. In addition, residual compressive stresses, which further improve the strength, are also introduced due to the differences in the coefficient of thermal expansion of the alumina and glass [5–9]. To enhance esthetics the framework is veneered with a feldspathic ceramic. The combination of this two-step process gave this framework material its properties, including a flexural strength that was three times the value of any other dental ceramic in the early 1990s, ranging from 236 to 600 MPa [10–16], and fracture toughness values ranging between 3.1 and 4.6 MPa·m^{0.5} [17,18]. As expected, such high values resulted in great clinical success, with one of the first studies reporting no failures out of 61 complete coverage crowns during a 35-month observation period [19].

Therefore, the success of the In-Ceram Alumina system and its patent expiration (April, 2007) instigated the release of similar ceramics from other manufacturers. Yet, none of these systems were investigated but are commercially available. The purpose of this study was to characterize the microstructure, composition, and important mechanical and physical properties of glass-infiltrated, alumina-based ceramic materials similar to the In-Ceram Alumina system, testing the null hypothesis that they all present comparable properties.

2. Materials and methods

The materials used were as follows: **IA**- In-Ceram Alumina (Vita); **IAE**- In-Ceram Alumina (Vita) electrophoretically deposited; **AEM**- In-Ceram Alumina (Vita) using a vacuum driven method; **VC**- Vitro-Ceram Alumina (Angelus, Londrina, PR, Brazil); **TC**- Turkom-Cera (Turkom-Ceramic, Kuala Lumpur, Malaysia); **CC**- Ceramcap (Foto-Ceram, Catalão, GO, Brazil); and **AG**- Alglass (EDG, São Carlos, SP, Brazil). All ceramic specimens (25 mm × 4 mm × 1.2 mm) were fabricated following manufacturers' instructions and ISO6872 standard using the traditional slip casting technique, except for the IAE and AEM, where the IA slurry was applied, respectively, by electrophoretic deposition (CeHa White ECS, C.

Hafner GmbH Co., Pforzheim, Germany, serial n.710B0008) and by a vacuum driven procedure (prototype machine, Vita Zahnfabrik, Bad Sackingen, Germany). All specimens were sintered and glass infiltrated according the manufacturers' instructions and allowed to cool. The excess glass was removed by sandblasting. All specimens were ground to 10 μm finish and were subsequently polished through 1 μm alumina abrasive (Mark V Laboratory, East Granby, CT, USA), sonically and steam cleaned using distilled water and examined for flaws using light microscopy (microscope model SCW30L, Fisher Scientific, Thailand) before performing the analytical analyses and flexural testing.

The dynamic elastic modulus (E) and Poisson's ratio (ν) were determined by means of ultrasonic waves and a computer program (ECALC and Sigview-F software, Nusun Inc., Boalsburg, PA, USA) based on a set of equations that uses the time of flight (TOF), density (ρ), and thickness (t):

$$\nu = \frac{TOF_s^2 - 2TOF_L^2}{2(TOF_s^2 - TOF_L^2)} \quad (1)$$

$$E = \frac{\rho t}{TOF_L^2} (1 + \nu)(1 - 2\nu)(1 - \nu) \quad (2)$$

The subscripted letters are longitudinal (L) and shear (s).

The specimen thickness (t) was measured using a digital caliper (Digimatic caliper, Mitutoyo Co., Kawasaki, Japan) and the weight (w) of the specimen was obtained using an analytical balance (Mettler H31, Mettler Instrument Corp., Hightstown, NJ, USA). The density ($n=5$) of the material was obtained using a helium pycnometer (Accupyc 1330, Micromeritics Instrument Co., Norcross, GA, USA) after calculating the volume (V). Piezoelectric transducers/receivers (Ultran Laboratories Inc., Boalsburg, PA, USA) and an ultrasonic pulse apparatus (Ultima 5100, Nusun Inc., Boalsburg) were used to measure the time of flight through the ceramic specimens. Longitudinal and shear (transverse) time of flight values were obtained and used to calculate the ν and E of the ceramics ($n=5$).

Semi-quantitative and qualitative analyses were performed using scanning electron microscopy (SEM), backscattered imaging (BSI), electron dispersive spectroscopy (EDS) and stereology principles. Polished ceramic specimens were mounted on aluminum stubs using carbon coating paste for better conductivity and were carbon coated in a vacuum evaporation system (Ion Equipment Co., Santa Clara, CA, USA). The thickness of the carbon layer was approximately 250 Å. Secondary electron and backscattered images (SEI and BSI) were recorded. The composition of each ceramic phase and overall composition of all ceramics were determined by electron dispersive spectroscopy (EDS) using a scanning electron microscope (JSM-6400, Jeol LTD., Tokyo, Japan) and the ZAF correction method. The analysis was performed at an accelerating voltage of 15 kV. Each ceramic phase was analyzed three times at different locations and average values of composition were determined. Compositional element concentrations above 1wt% were recorded.

The crystalline volume fraction (V_v) was estimated based on stereological principles [20]. An auto beam-area measurement function was used based on intensity histograms from backscattered electron images (Link ISIS program, Oxford, England and Link ISIS Instruments, Microanalysis Group, Bucks, England). Eight different areas from three specimens were used and averaged for the V_v value.

Ceramic specimens (n=20) were loaded in three-point bending to fracture using a universal testing machine (EMIC DL2000, São José dos Pinhais, PR, Brazil) with cross-head speed of 1mm/min. The cross sectional area of each ceramic specimen was measured (Digimatic caliper, Mitutoyo Co., Kawasaki, Japan) and used to calculate the flexural strength (σ_{3P}). Weibull regression analysis was performed on the strength data as previously described [21] to obtain the Weibull modulus (m) and the characteristic strength (σ_0) at 95% confidence interval.

A microhardness tester with a Vicker's indenter (Micromet 3, Buehler, Lake Bluff, IL) was used to indent the polished surface of the fractured bar-shaped ceramic specimens (n=10) and calculate hardness (H) following the ASTM C1327-03 standard, with a load (P) of 19.6 N and a dwell time (t) of 15 s.

One-way analysis of variance (Anova), Tukey at 5% significance level ($\alpha=0.05$), and Pearson correlations were applied to some of the properties for statistical purposes.

3. Results

The mean and standard deviation values for the properties investigated in this study are presented in Table 1.

The semi-quantitative compositional analyses (average %) by EDS (excluding the C and O elements) of the In-Ceram Alumina specimens fabricated by different methods (**IA**: traditional slip casting; **IAE**: electrophoretic deposition; and **AEM**: vacuum driven) resulted in very similar composition: 12% La(L); 83% Al(K); 4% Si(K); and 1% Ca(K). The average overall composition of the other ceramics were as follows: **VC**- 11% La(L), 74% Al(K), 5% Si(K), 4% Zr(L), 5% Y(L), and 1% Ca(K); **TC**- 17% La(L), 76% Al(K), 5.5% Si(K), 0.5% Na(K), and 1% Ca(K); **CC**- 50% Pb(M), 45% Al(K), 4.5% Si(K), and 0.5% Na(K); and **AG**- 15.5% La(L), 79% Al(K), 3.5% Si(K), 1% Ca(K), 0.7% Na(K), and 0.3% K(K). The most important compositional differences are the presence of Zr and Y elements in the VC ceramic, and the substitution of Pb for La in the CC ceramic. A specific analysis revealed an average of 84% of Pb in the glassy phase of CC ceramic.

SEM and EDS analyses showed similar microstructures for all glass-infiltrated alumina-based ceramics, except for the VC ceramic with zirconia as a second crystalline phase. In addition, it appeared that the alumina crystals were more compacted in the AEM, which was confirmed by the lowest percentage of porosity (Fig. 1).

The volume fraction (V_v) of the alumina phase is presented in Table 1. The EDS-Link ISIS analysis showed that VC ceramic has about 3% of zirconia (Fig. 1). The average porosity was 7%, ranging from 4% to 20%, respectively, for AEM and AG. The greatest porosity of AG resulted in the least mean strength and density values, and impaired the H measurements in such a way that many indentations were needed to acquire the requisite sample size. The amount of glass ranged from 17% to 51%, respectively, for the Vita ceramics (IA, IAE and AEM) and CC.

The mean values of Poisson's ratio and density were similar for all ceramics, with a small standard deviation (≤ 0.02).

The mean flexural strength values of IA, IAE, AEM and VC were not statistically different ($p>0.05$). The AEM ceramic showed the greatest m (16) and E (300 GPa).

Some of the properties were positively correlated as follows: σ_{3P} and E ($r=0.84$); σ_{3P} and H ($r=0.94$); σ_{3P} and V_v ($r=0.76$); E and H ($r=0.87$); E and V_v ($r=0.89$); H and V_v ($r=0.86$); σ_{3P} and porosity ($r=0.8$).

4. Discussion

Glass-infiltrated ceramics have been in clinical use as core materials for all-ceramic restorations for almost 20 years [5,10,11,19,21–24]. The distinct advantage of these ceramics is the easy near-net-shape forming, yet a high strength in the infiltrated state [25]. The In-Ceram system was developed using an alumina slip casting technique to build the framework that is fired to form an open-pore microstructure, which is infiltrated with lanthanum oxide-based glass. Therefore, the values indicating the high mechanical properties of this system depend upon the complete wetting of the porous microstructure by glass infiltration [21]. Many “In-Ceram like products” were made commercially available with the expiration of the Vita In-Ceram patent in 2007. However, there is no report on these ceramics properties, which impairs any estimation of their clinical performance.

Microscopic examination is useful for the study and characterization of materials. Material microstructures are often related to material properties, and the information may be used to predict properties and improve the design of new materials [3,21].

There are important relationships between chemical composition, atomic structure, fabrication process, microstructure, and properties of polycrystalline ceramics. The role of the fabrication process, for example, is to produce microstructures with desired chemical characteristics and properties. Each processing step has the potential for producing undesirable microstructural flaws in the ceramic body that can limit its properties and reliability. Thus, the microstructure, which refers to the nature, size, shape, quantity, and distribution of the structural elements or phases in the ceramics, has a profound effect on physical properties. Therefore, quantifying microstructural parameters is important to develop structure/property relationships. Quantitative microstructural analysis provides an association among the constitution, physical properties, and structural characteristics of materials [3,21].

Several methods can be used to determine ceramic composition. This study employed a semi-quantitative analysis using electron dispersive spectroscopy (EDS), according to the protocol suggested by Della Bona and Anusavice [3]. According to those authors, this is one of the more reliable methods to analyze the composition of different ceramic phases.

Currently, to fabricate the In-Ceram framework, the ceramist can either mill out a dry-pressed partially sintered block or use the slip-casting technique. Previous studies on IA indicate that the microstructure of the slip-cast material is coarse and heterogeneous, having about 20% of glass and 75% of alumina [23,25], which is in accordance with the results of the present study (Fig 1 and Table 1). The microstructure of IA and IAE were very similar, yet different from AEM. All three ceramics used the same slurry from Vita In-Ceram Alumina but the processing methods were different. It seems that the vacuum driven experimental method (AEM) resulted in a more compacted microstructure, which was confirmed by the reduced amount of porosity. The other ceramics showed few differences in their microstructure and composition, possibly reflecting differences in the size and shape distributions of the starting powders. The VC showed about 3% of zirconia as a second crystalline phase, characterizing this material as a glass-infiltrated alumina-based zirconia-reinforced ceramic, similar to In-Ceram Zirconia but with much less vol% of zirconia [21]. The compositional analysis of CC showed about 51% of a lead-based glass, which should decrease the ceramic glass transition temperature (T_g), increase the expansion of the glass

and reduce the glass viscosity, which would be beneficial for infiltration. These results were sent to the Brazilian Regulatory Agency (Anvisa), and the material (CC) was removed from the market.

The mean E and H values of TC, CC and AG were statistically less than for the other ceramics ($p < 0.05$). The mean values of E, H, and Vv for IA are in agreement with a previous report [25]. VC showed similar mean E and H values as IA and IAE ($p > 0.05$), but a lower Vv for the alumina phase, when compared with the ceramics IA, IAE and AEM.

This is mainly due to the presence of a second crystalline phase (zirconia) and a greater glass content, which is about 27% for VC compared to the 20% of the Vita ceramics (IA, IAE, and AEM). The basis for the proper design of the connectors and the pontic is the law of beams: the maximum deflection of a beam increases as the cube of its length, it is inversely proportional to its width, and it is inversely proportional to the cube of its height. A 3-point bending test is one of the most commonly used tests to determine the modulus of rupture, or the transverse flexural strength, of a rectangular beam made of a brittle material [16,26]. When an occlusal load is applied through the vertical axis of an all-ceramic FPD at the midspan (the pontic in a three-unit FPD), compressive stresses develop at the occlusal part of the connector, and tensile stresses develop at the gingival surface of the connector [27]. The tensile stresses contribute to the propagation of critical defects located at the gingival side of the connector through the core material in an occlusal direction, eventually resulting in catastrophic failure of the restoration [28]. This rationale was the reason for using the flexural strength testing and the Weibull distribution for analysis of the data.

The mean σ_{3p} and σ_0 values for IA, IAE, AEM and VC were not statistically different ($p > 0.05$), respectively. However, the mean m value of AEM was greater than the other ceramics. These high mean σ_{3p} , σ_0 , and m values for AEM, which is the Vita In-Ceram ceramic processed using the experimental vacuum driven method, can be explained by the lowest porosity (4%). Based on knowledge of general behavior of ceramics, we would expect that low porosity would result in greater elastic modulus and greater toughness. These increased properties then would be expected to result in greater strengths. Despite the differences in microstructure and composition among the ceramics examined in the present study, the mean flexural strength values are in agreement with previous studies that report values ranging from 236 MPa to 600 MPa [11–15].

Positive correlations were revealed between the main properties investigated (E, H, Vv, and σ_{3p}). These results confirmed the expected material properties relationship.

As shown in this study, the glass-infiltrated alumina-based ceramics from different manufacturers presented distinct characteristics. It is necessary to characterize all new commercially available materials to understand their properties and estimate their clinical performance. As this study used monolithic core ceramic specimens, the effect of flaws in the veneering ceramic were not taken into account. This is a limitation of the present study since these flaws may influence the strength of the connector area of ceramic restorations. Future studies should investigate the fracture toughness and the dynamic fatigue of the new ceramic materials including multilayer ceramic structures.

5. Conclusion

Despite a few differences in microstructure and composition, the IA, IAE, AEM and VC ceramics have similar properties. The TC and AG ceramics showed poorer characteristics and properties than the Vita ceramics. The CC ceramic was withdrawn from the market because of its poor quality and inadequate composition.

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Figure 1.

Representative images (SEM in BSI mode) of carbon coated ceramics; the dark crystals are alumina in the glassy phase (white to light grey). The VC image has a third phase of light crystals (zirconia). The images are colored by the ISIS software for volume fraction calculations. An intensity histogram is produced from the percentage values of red (glass) and green (alumina) as measured by the Link ISIS computer program, except for VC where red is zirconia, and black is the glassy phase. In all other colored images (1B), black indicates porosity. Magnification is 5000 $\times$ (For interpretation of the references to color in this figure legend, the reader is referred to Fig. 1B in the web version of the article).

Table 1

The mean and standard deviation values and the statistical groupings (*) of flexural strength (σ_{3P}), characteristic strength (σ_0), Weibull modulus (m), elastic modulus (E), Poisson's ratio (ν), hardness (H), density (ρ), and the volume fracture (V_v) of the alumina phase (Al_2O_3).

	σ_{3P} (MPa) *	σ_0 (MPa)	m	E (GPa) *	ν	H (GPa) *	ρ (g/cm ³)	V_v (% Al_2O_3)
IA	489±62a	505±16	9±4	280±4b	0.25	12.1±1.1a	3.8	74±3
IAE	485±63a	516±15	9±2	277±3b	0.26	12.9±1.1a	3.8	75±3
AEM	517±37a	528±8	16±5	300±4a	0.25	11.3±0.5b	4.0	72±3
VC	529±67a	560±12	9±3	284±5b	0.24	12.4±0.4a	3.8	62±3
TC	332±44b	352±5	8±2	245±3c	0.25	9.3±0.7c	4.2	63±3
CC	258±28c	271±6	9±2	166±1d	0.23	7.4±0.5d	4.2	42±2
AG	233±26c	244±8	9±3	239±2c	0.25	8.5±0.8c	3.6	57±2

* Similar letters in the column show no statistical difference ($p>0.05$).