Influence of handling and heating cycle on the morphology and flexural strength of glass-infiltrated alumina bars

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ABSTRACT

The main goal of the present study was to evaluate the influence of plaster stability, handling and thermal cycling on the morphology and mechanical strength of glass infiltrated alumina bars. Two different slip casting/thermal processing routes were investigated. The highest flexural strength of alumina bars from In-Ceram® Alumina protocol in comparison to In-Ceram® Sprint protocol was mainly due to its low drying rate and less handling. After glass infiltration and mechanical surface treatments, In-Ceram®Sprint alumina bars presented a non-homogeneous distribution of alumina particles in the glass phase, which was the mais cause for its lower flexural strength. In both cases, it was observed a strong anisotropic growth of alumina particles after glass infiltration. Non-reproducible mechanical surface treatments is other factor that could deteriorate the mechanical strength of alumina bars.

Key-words: Glass infiltration, alumina bars, flexural strength.

1   INTRODUCTION

Due to its high mechanical strength and biocompatibility, dental ceramics is one of the main choices for crowns or fixed partial dentures [1, 2]. However, its low fracture toughness limits its applications, in comparison to metals or special alloys [3]. In order to increase its uses and reduce costs, porcelains over metal named metal-ceramics, has been used extensively for the last three decades. Actually, the main aim for dental prosthesis is substituting the metallic substructure for a ceramic one, which allows high strength, aesthetics and biocompatibility [4].

A method for making all-ceramic dental prosthesis having high strength and low shrinkage was patented by Tyszblat,M. [5] which consisted of a partially sintered alumina framework further infiltrated by a glass phase. One of the most interesting aspect of this process, is its near-net shape processing. Based on this route, VITA Zahnfabrik patented two different routes called In-Ceram®Alumina and In-Ceram®Sprint. In both cases, an alumina crown is prepared by brushing a 65 vol% alumina slip on the surface of a special plaster mold, followed by partial sintering and glass infiltration. It is important to point out that handling and heating cycle of both routes are significantly distinct.

Mechanical surface treatments of those glass infiltrated frameworks are commonly used to remove the excess glass and to make size adjustments. The influence of grinding on its mechanical strength are contradictory [6, 7]. It can be explained by taking in account the combined effect of surface flaws and residual stresses, the ratio of crack length to surface compression layer depth, the dimensions of pre-existing flaws and other factors. In spite of its low impact energy, sandblasting tend to create some strengthening and micro retentive regions in its surface increasing cement adhesion to the framework [8, 9]. Polishing tend to promote some strengthening based on a similar patented mechanism as sandblasting, that is by generating surface compressive stresses and by removing pre-existing flaws or radial cracks.

The flexural strength of alumina bars from both routes range between 240 and 600MPa [10-13]. This high mechanical strength can be explained by the high Young modulus of alumina and by the crack bridging mechanism of alumina and the glass phase, where friction dissipates the energy of crack

propagation [12]. Clinical performance of In-Ceram Alumina is relatively scarce and some contradictory. Sorensen et al. [13] recommend that this system could be used with confidence for anterior fixed partial dentures and patented von Steyern et al. [14] reported a 90% success of three unit posterior fixed partial denture of In Ceram Alumina after five years.

2   MATERIALS AND METHODS

The chemical composition of alumina and infiltration glass frit powders determined by X-ray fluorescence, are shown in table 1. The alumina particle size varied between 2.5-10.0 μm with some submicrometric. The morfology of alumina was basically isotropic.

Table 1: Chemical composition of alumina and infiltration glass frit powders

Alumina	Weight %
Al_2O_3	99.70
MgO	0.03
Infiltration Glass Frit	**Weight %**
La_2O_3	49.60
Al_2O_3	20.00
SiO_2	19.10
TiO_2	6.16
CaO	3.14
Others	2.00

In order to prepare a well dispersed concentrated alumina slip (38g Al_2O_3 in 5ml aqueous solution) at room temperature, it was necessary to identify if in this conditions the alumina particles were well deffloculated. The stability of alumina slip was confirmed by the zeta potential X pH graph. As shown in figure 1, the zeta potential of alumina slip at pH (pH = 6.8) was approximately + 80m, indicating   well dispersed conditions.
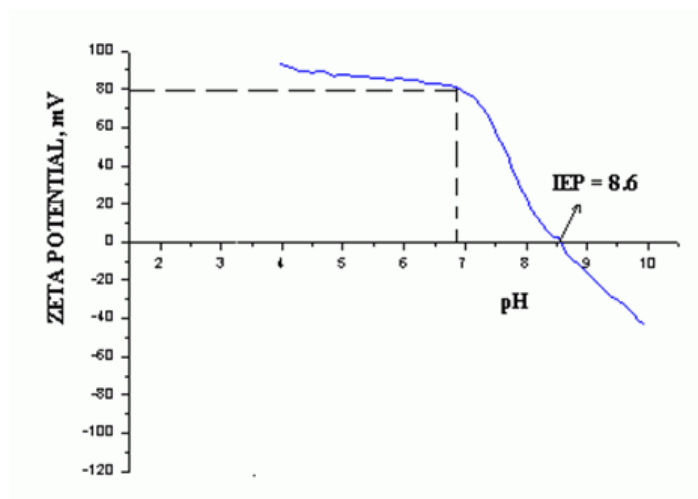

Figure 1: Zeta potential X pH of alumina in aqueous solution. IEP – Isoelectric point of alumina

The alumina bars were molded (25 x 4 x 1.2 mm) on a stainless steel matrix over a plaster plate, as shown in figure 2. Figure 3-a shows the microstructure of the plaster plate at the end of drying and figure 3-b at the end of   partial sintering heating cycle. It is important to comment that only in In-Ceram Sprint processing, the alumina bar was removed from the plaster mold after drying and re-introduced in the furnace

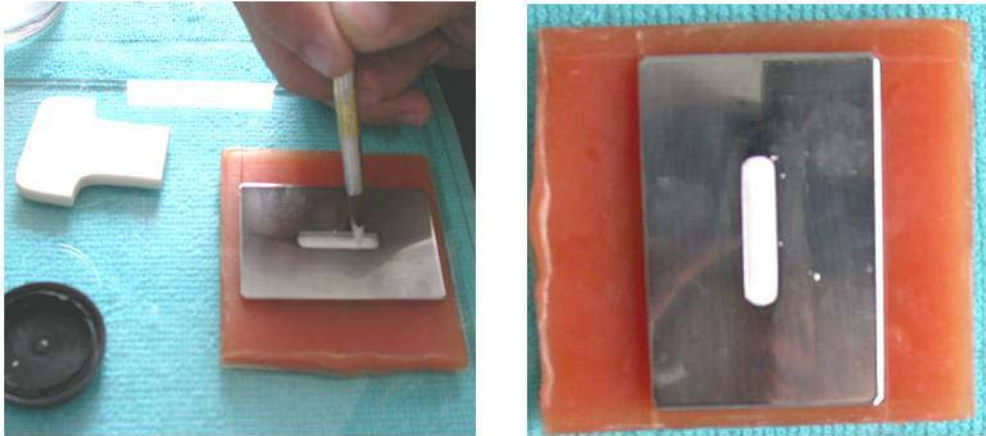to complete its heating cycle. Figure 4 shows In-Ceram[®]Alumina and In-Ceram[®]Sprint partial sintering cycles.

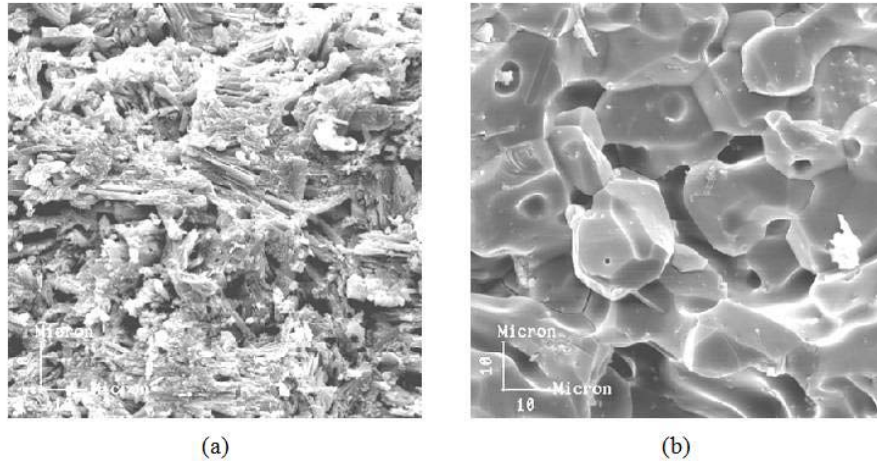**Figure 2**: Molding alumina bar by slip casting over a plaster mold

Figure 3: (a) Microestruture of plaster plate at the end of drying;
(b) Microestruture of plaster plate after partial sintering

The pore size and pore size distribution of alumina bars partially sintered were determined by a mercury porosimeter (Quantachrome Autoscan 3.0). In both routes (In Ceram Alumina and In Ceram Sprint), the pore size distribution was very similar   (0,05-0,5μm). The glass infiltrated alumina bars $(1100^{0}C/120$ minutes followed by slow cooling inside the furnace), did not show any residual porosity.
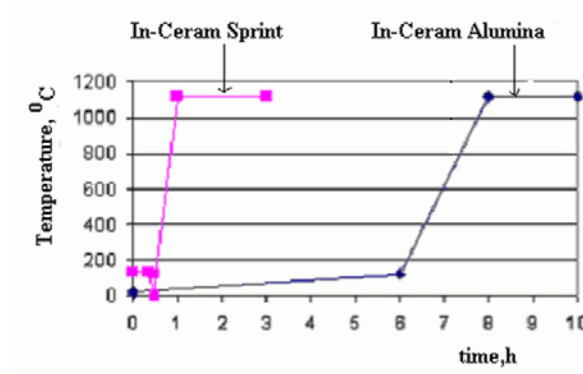
Figure 4: In-Ceram®Alumina and In-Ceram®Sprint partial sintering heating cycles

In order to remove the excess glass and to adequate the alumina bars dimensions to the ISO 6872 standard, ten bars of each processing route were grinded, sandblasted and polished. Figure 5 shows the alumina bar being tested in the equipment EMIC MFDL 500 (load cell of 500N and crosshead speed 0,5 mm/min). The reliability of flexural strength data was evaluated through Weibull statistics.

(a)   (b)

Figure 5: (a) Flexural test equipment EMIC MFDL 500. (b) Detail of the alumina bar being tested (b)

3   RESULTS AND DISCUSSION

Infiltrated alumina bars presented a mean flexural strength (555MPa) higher than In-Ceram Sprint (430MPa). This behavior was mainly due to handling and to the different heating cycles. The high drying rate and handling in In Ceram Sprint route, are probably the main causes for its lower strength. Figure 6 shows the Weibull plot for the flexural strength of In-Ceram Alumina bars. These results indicate some scattering of experimental data (m = 6.5). The flexural strength corresponding to 63% cumulative probability of failure was 600MPa.
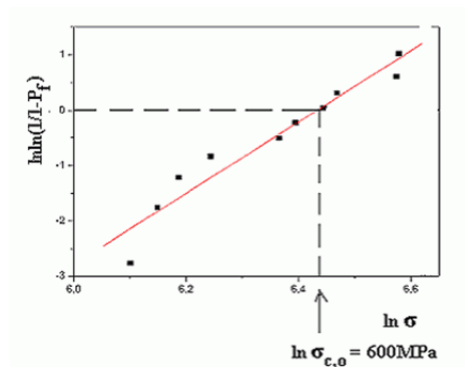

Figure 6: Weibull distribution of infiltrated alumina bars (In-Ceram Alumina)

Figure 7 shows the scanning electron microscopy of an alumina bar before and after glass infiltration. The anisotropic growth of alumina after lanthanum glass infiltration is clearly seen. The lanthanum glass does not have the classical network modifiers (alkaline and alkaline-earth), but in this case La_2O_3 act as a glass modifier decreasing its viscosity.[34 (raquel)]. These glasses have interesting mechanical properties, for example high elastic modulus, hardness and glass transition temperature (900^0C). After polishing, as shown in figure 8, In-Ceram Sprint alumina microstructure presented a higher volume of infiltrated glass, which was probably related to particle size distribution and to the deffects generated during drying and handling. It is expected that these are the main causes for the lower mechanical strength of In-Ceram Sprint alumina bars.
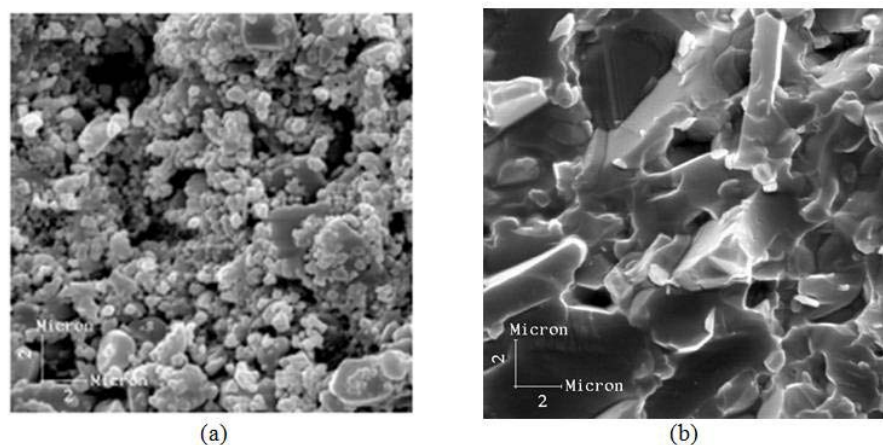
Figure 7: Scanning electron microscopy of fractured non-infiltrated (a) and an infiltrated alumina bar (b). Observe the anisotropic growth of alumina after glass infiltration.. Bar length - 2μm
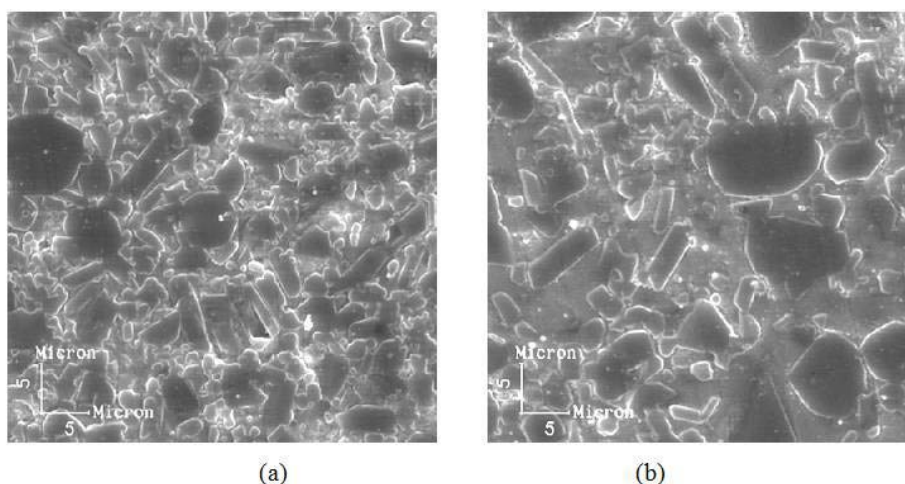

Figure 8: Scanning electron microscopy of polished glass infiltrated alumina bars.
(a) In-Ceram®Alumina , (b) In-Ceram® Sprint

Compairing the mechanical strength, pore size distribution and microstructure of infiltrated alumina (In-Ceram® Alumina and In-Ceram® Sprint) with the one from Lee,D.Y. et al. [15], it was observed that they were very similar. Guazzato et al. [16] reported a slightly higher mean flexural strength of In-Ceram® Alumina (594MPa). The influence of different mechanical surface treatments (grinding, sandblasting and polishing) on the mechanical strength of glass-infiltrated alumina dental ceramic was reported by Guazzato,M. et al.[6]. It was shown that grinding and sandblasting promote a severe damage on dental prosthesis mainly during excess glass removal. Despite the use of a water coolant, Swain and Hannink [17] dealing with zirconia, observed that an excessive heat generated during grinding could promote changes in its microstructure. In this case, this heating could promote crack healing which did not occurred during sandblasting.

It is important to point out that in both routes, it was observed a strong anisotropic growth of alumina after glass infiltration (figure 8). The capillary glass flow through the open porosity of partially sintered alumina and the solution/precipitation of alumina in the glass phase are the main causes for its high anisotropic growth. Guo et al [18] suggest that solid state diffusion of La^{3+} in Al_2O_3 at high sintering temperatures may form needlelike $LaAl_{11}O_{18}$. Maciel,A.P et al. [19] also observed needlelike formation of $LaAlO_3$ from the solid state reaction between La_2O_3 and Al_2O_3 of at approximately 1400^0C. In the present work, in spite of the lower glass infiltration temperature (1100^0C), a possible diffusion of La^{3+} in Al_2O_3 lattice might occur, mainly due to the wetting of alumina particles by the glass phase. In this case, solution-precipitation mechanism as proposed by German [20] could occur. If this mechanism is operative, the dissolution of alumina in the glass phase from high stress regions and precipitating at low stress regions could be the most feasible.

4   CONCLUSIONS

Due to the low drying rate and less handling, In-Ceram®Alumina (group 1) presented the higher mean flexural strength and reliability;

Due to its high drying rate and relatively impervious plaster mold due to the insulation gel film, In-Ceram Sprint present a more opened structure; the glass phase penetrating in a great amount in the porous alumina promote a significant decrease in mechanical strength due to the lower strength of the glass phase;

The flexural strength of all sample groups were influenced by mechanical surface treatment for excess glass removal. The high dispersion of flexural strength data of both groups were mainly due to glass infiltration and mechanical surface treatments for excess glass removal;

The strong anisotropic growth of alumina particles during glass infiltration was mainly due to the interaction between alumina and the liquid phase. The solubility of alumina in the glass phase and the influence of temperature   on its solubility,   may possibly explain the impressive anisotropic growth of alumina particles.

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