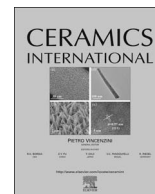




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Failure analysis of glazed LAS glass-ceramic containing cerium oxide

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ABSTRACT

In this study a deep investigation of the correlation between microstructure and mechanical properties was performed on glass-ceramic materials containing cerium oxide. The glass-ceramic materials investigated were lithium-aluminum silicate based (LAS), obtained through different thermal cycles from the same parent glass. The mechanical properties were tested on the glass-ceramics with and without the application of a glaze. All the glass-ceramics were characterized by means of scanning electron microscopy (SEM), also in field emission guns geometry (FEG), energy-dispersive spectrometry (EDS), X-Ray diffraction (XRD), and 3-points mechanical flexural strength resistance test. This study has revealed that the microstructure of the glass-ceramic is mainly affected by the heating treatments holding times. The application of a glass coating leads to the formation of cerium oxide crystals at the glaze/glass-ceramic interface. This phenomenon is one of the main principal causes of the mechanical properties failure when a glaze is applied.

1. Introduction

Lithium aluminum disilicate (LAS) glass-ceramic is one of the most important glass-ceramic systems because of high thermal and chemical durability. In fact LAS glass-ceramics are used in several industrial fields to produce cookware, stovetops, fireplace windows and dental prosthesis [1–4].

LAS glass-ceramic system is part of β -spodumene solid solution glass-ceramic group, $\text{Li}_2\text{O Al}_2\text{O}_3 \cdot n\text{SiO}_2$ where $4 < n < 10$ [5]. One of the others components that commonly is founded in this glass-ceramic system is P_2O_5 , that plays a crucial role in crystallization [6,7]; in fact, P_2O_5 as heterogeneous nucleating agent at the amount of 1.5–2.5 mol %, promotes volume nucleation of the lithium silicate phases, conferring high mechanical properties to the final products [8,9]. Moreover, coloration can be easily achieved by addition of V3+(green), Ce4+(yellow) oxides to the raw powder [9]. Finally in β -spodumene solid solution glass-ceramic group CeO_2 , TiO_2 and ZrO_2 , can act as nucleant agent [5]. Considering in particular the cerium oxide influence on LAS glass-ceramic microstructure, it is possible to find out that cerium oxide in low quantity, up to 5%wt, promotes crystallization [3], but also flexural strength decrease and thermal expansion coefficient increase [10]. In fact, cerium oxide addition enhances the growth of the glass-ceramics grain, but larger are the grain boundaries highest is the probability to break the microstructure, because glass-ceramic can be easily broken along the grain boundaries.

For LAS glass-ceramics the preferential crystallization and nucleation mechanism is the one of volume, however, surface reactions

cannot be neglected, when considering nucleation and crystallization in powder compacting and subsequent sintering [11].

From a suitable parent glass, ceramic-glass is obtained through heating treatment, during which crystallization occurs. The typical mechanism of crystallization is a volume crystallization where the crystals nucleate and grow throughout the glass. Crystallization of lithium disilicate is heterogeneous and can be achieved through different processing routes. Considering one of the most commons process [9], the parent glass is formed into glass blocks or by pressure casting into a steel mold, starting from a glass melt of synthetic raw materials. Before cooling down to room temperature, the poured melt is transferred into a pre-heated furnace at 450–550 °C to avoid stress in the glass block. The subsequent crystallization is carried out in a three-stage crystallization process, in order to precipitate lithium disilicate. In the first stage, the glass block is heated at 450–550 °C for about 1 h to form the maximum number of nuclei to ensure the crystal growth of metasilicate crystals. In the second stage, the glass block is heated at a temperature of 690–710 °C for 10–30 min to form lithium metasilicate (Li_2SiO_3) crystals, grown epitaxially from the lithium orthophosphate nuclei. This intermediate metasilicate phase is present in a volume fraction of 40%. The glass-ceramic at this stage is still weak, but thanks to its properties it can be milled to obtain a specific geometry. In the third stage the material is heated at 850 °C for 20–30 min in a furnace to precipitate the final strong lithium disilicate phase and some minor amounts of lithium orthophosphate crystals. During this stage, the lithium metasilicate crystals react with the glass matrix to form lithium disilicate in a volume fraction of up to 70%. A

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further process commonly applied in several field is glazing. Glazing, that is the application of glass coating on the surface of the glass-ceramic, is commonly employed in order to increase the chemical durability of the material and to enhance a specific coloration [12]. In fact, glazing techniques can avoid the effects left by sandblasting and polishing in order to obtain sealed hygienic surfaces [13]. On the other hand glazing process has been investigated because of its influence on the cracking phenomenon [14].

The aim of the present study is to investigate the influence of the heating thermal cycle and the application of a glaze coating on the mechanical flexural strength of a series of LAS glass-ceramic. The properties of the different LAS glass-ceramic were determined through scanning electron microscopy (SEM), also in field emission gun geometry (SEM-FEG), energy dispersive spectroscopy (EDS), 3-points flexural strength resistance, X-Ray diffraction (XRD) and Rietveld refinement.

2. Materials and methods

2.1. Glass preparation

A parent glass of the LAS system with the composition of 66%SiO₂, 6%Al₂O₃, 22%Li₂O, 1%CeO₂, 5% others oxides in mol% was used in this study. This glass was melted in platinum crucibles at 1550 °C. Reagent grade raw materials were used. After a holding time of 1 h at 1550 °C the glass was poured in a rectangular graphite mold and then transferred into a pre-heated furnace at 450–550 °C to avoid stress in the glass block. From a block of this glass, specimens with approx. 5 mm×5 mm×40 mm were cut and polished. Polishing was done with SiC discs, the sequence of the discs was: P80, P400, P800, P1000 and P2500, according to FEPA-Standards 43–1:2006, 43–2:2006 and in ISO 6344. The grinding time was 5 min for each specimen-side and disc. The load was applied by finger pressure and manual polishing cooled by water was performed in small circles. Each sample of glass was polished to avoid any difference of roughness on the surfaces and to correct the shape, in order to obtain the most possible regular geometry and same smoothness surface.

2.2. Glaze preparation

Glass with the composition of 69%SiO₂, 10%B₂O₃, 3%Al₂O₃, 2%Li₂O, 2%CaO, 4%Na₂O, 4%K₂O, 1%CeO₂, 5% CaF₂, in mol%, was chosen as the optimized glass for this purpose, as reported in a previous study [15]. This glass was melted in a batch of 25gr in platinum crucibles at 1500 °C. Reagent grade raw materials were used. After a holding time of 1 h at 1500 °C the glass was cast into water, dried and crushed in agate mill to obtain a fine powder. This frit was mixed with a commercial liquid glycol-water based to obtain a suitable slurry of glaze.

2.3. Specimens preparation

The parent glass specimens were employed to perform different heating cycle tests, heating rate 10 °C/min, according to Tables 1, 2. The specimens preparation has been performed in two phases, concerning the effect of the glazing process or not.

In the first set of data only the heating cycle from parent glass to glass-ceramic was studied (Table 1). In the second set of data the effect of the glazing process was considered (Table 2).

Considering Table 1, the heating cycle tests were chosen from a common heating process [9], named C0, according with the DSC measurement as shown in Fig. 3. The following test, from C1 to C5 were chosen to evaluate the effects of a slight difference in temperature or time from C0 on to the mechanical properties. Whereas C6 and C8 conditions were selected to enhance two opposite behavior in terms of holding time of the heating cycle from C0. On the other hand,

Table 1

Heating cycle tests for not glazed specimens.

	Nucleation		Growth		Final	
	T (°C)	t (min)	T (°C)	t (min)	T (°C)	t (min)
C0	540	300	660	30	830	90
C1	490	300	610	30	830	30
C2	590	300	710	30	830	90
C3	460	300	580	30	830	15
C4	540	300	660	30	830	120
C5	620	300	740	30	830	90
C6	540	600	660	600	830	120
C8	540	150	660	15	830	15

Table 2

Heating cycle tests for glazed specimens.

	Nucleation		Growth		Final		Glaze
	T (°C)	t (min)	T (°C)	t (min)	T (°C)	t (min)	
C7	540	600	660	600	830	120	YES
C9	540	150	660	15	830	15	YES

considering Table 2, glaze was applied on both the more extreme conditions in terms of holding time of the heating cycle, generating test C7 and C9, in order to investigate the effect of glazing on two very different conditions in terms of heating cycle.

For each test, 6 pieces, selected randomly, were employed to have a relevant statistic.

For test C7 and C9 the glaze was applied manually with a brush on one of the larger surface of the specimens after the 3-stage heating cycle. The glazed samples were dried at 120 °C for 4 h and fired at 800 °C for 5 min (heating rate 20 °C/min). The cooling of all the specimens was performed into the furnace until room temperature. The glazing heating cycle conditions were fixed in order to reduce the number of variables.

2.4. Characterization of the samples

In this study, the parent glass was analyzed by using a X-Ray Diffraction (XRD, Philips, PW3710) and scanning electron microscopy (SEM, FEI XL-30) in order to verify any presence of crystalline structures inside the glass. Furthermore the thermal behavior of the parent glass was measured using DSC analysis (Netzsch, DSC 404) performed in air at the heating rate of 10 °C/min using 30 mg of the powdered sample. The thermal behavior of the parent glass was analyzed in order to plan the heating cycle tests, from parent glass to glass-ceramic.

A thermo-mechanical characterization was made on the glaze and on all the glass-ceramic samples, through an optical dilatometer (Expert System Solutions, Misura ODHT) at the heating rate of 10 °C/min until 550 °C, in order to obtain the linear coefficient of thermal expansion (CTE- α) [16]. In order to identify the characteristic points of sintering, softening, sphere, half-sphere and melting, a hot-stage microscopy analysis was performed on the glaze (Expert System Solutions, Misura HSM) at the heating rate of 10 °C/min until 1200 °C [17]. The thermo-mechanical properties of the glaze were analyzed to compare the CTE- α of the glaze with the CTE- α of the glass-ceramic support. The glaze was also investigated with X-Ray Diffraction (XRD, Philips, PW3710), in order to verify the presence of any crystalline phase.

The 3-points flexural strength of each LAS glass-ceramic specimen was examined using the apparatus INSTRON 5567 in order to measure

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