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Research article

Development of cordierite-based glass-ceramics by slip casting through selecting the appropriate sintering conditions

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A B S T R A C T

The present work aims to prepare a dense cordierite-based glass-ceramic through slip casting and consequent heat treatment procedures. In this regard, sintering conditions were considered as the key variables to improve the properties of the glass-ceramic. For this purpose, glass frit powder was prepared through melting oxide powders (in the system of SiO_2-Al_2O_3-TiO_2-K_2O-CaO-MgO). The mixed powders were then heat treated at 1450 °C for 1 hour and quenched in water. The glass frit powder was slip cast using the appropriate dispersant. Sintering was carried out by one-step, two-step, and three-step procedures. Specimens were characterized in terms of various analysis techniques including dilatometry, X-ray diffractometry, scanning electron microscopy, and mechanical strength measurement. Among the examined specimens, the sample sintered by a three-step approach was considered the optimized one which attained zero porosity. According to the obtained results, cordierite crystals were observable in this glass-ceramic matrix. A low coefficient of thermal expansion and a low dielectric constant were observed for the optimized glass-ceramic sample. The obtained results confirmed that the homogenous distributions of crystalline phases are responsible for the appropriate and desirable properties of the prepared glass-ceramic.

KEYWORDS

Cordierite
Glass-ceramic
Slip casting
Sintering

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1. Introduction

Cordierite ($2MgO.2Al_2O_3.5SiO_2$) and cordierite-based glass-ceramics have attracted much attention in recent years as electronic and radio engineering materials because of their low thermal expansion coefficient, low thermal conductivity, and low dielectric constant [1–3]. Therefore, cordierite has the potential to be used in protective radio equipment and microwave packaging [4–6]. To satisfy these specifications, cordierite glass-ceramics should be fully sintered with very low water adsorption.

Solid-state sintering of cordierite ceramics is difficult, and sintering aids should be used to provide liquid phase sintering in MgO-Al_2O_3-SiO_2 (MAS) system. The additives affect the electrical and thermal properties of these materials which are critical in some advanced applications [7]. It has been shown that the sintering process, as well as the processing route, particle size, particle packing, and the purity of raw materials, allows tailoring the properties of ceramics. Some researchers used the benefits of pressure, changing the composition, processing before firing, etc. to attain complete densification [8]. In this way, two-step sintering was used to prepare dense MAS glass-ceramics [1]. By controlling the sintering conditions, the thermal and dielectric properties of the cordierite-based ceramics can be improved.

Several methods have been studied for preparing cordierite glass-ceramics. The most common method is crystallization from the compacted glass powder. Slip casting of oxides or natural materials is also mentioned [8]. Slip casting is one of the most widespread methods for preparing ceramics, which is of interest to even recent papers [9]. Making of objects of any shape and uniformity can be attained through slip casting [6]. Akpinar [8] studied the rheological properties of cordierite glass ceramics prepared through slip casting of kaolin,

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magnesia, and silica powders. Mei et al. [7, 10] investigated the slip casting of cordierite powder. They mainly focused on the rheological behavior of the cordierite suspensions and aimed to attain a homogeneous microstructure. Although they concluded that slip casting is an efficient technique to prepare dense and homogeneous cordierite-based glass-ceramics, they preferred to use a glass powder as a sintering aid. There is a study on the slip casting of glass powder in the MAS system, however, they could not obtain the desired properties and their samples suffered from some amount of porosity, therefore, they tried to crystallize their glass powder and then casting [6]. Marghussian et al. [11] studied the slip casting of cordierite glass powder. They focused mainly on the rheological properties, suspension preparation, and the effect of pH; however, they did not attain full densification (about 95% of the theoretical density) or high strength (72 MPa). It has been also shown that cordierite glass-ceramics can be fabricated through slip casting; however, there were many pores and flaws in the prepared microstructure [4].

The main purpose of this study is to investigate the feasibility of preparing dense cordierite-based glass-ceramics by slip casting without the use of sintering aids. The advantages of the slip casting procedure were used through which complex and industrial shapes can be fabricated. However, dense cordierite glass-ceramics are hard to achieve [2]. Fabrication of dense cordierite-based glass-ceramics is the challenge of this paper which did not carry out properly. Preparing these samples through slip casting has not provided the required results so far. Therefore, it was aimed to prepare a sample with adequate strength and low adsorption which is suitable for advanced applications. The time and temperature of the heat treatment control the size, the number, and the rate of growth of the crystals [1]. Therefore, the different sintering conditions (multi-step sintering) were chosen as the main parameter and their effects were evaluated on the microstructure and physical properties of cordierite-based glass-ceramics.

2. Experimental procedures

Silica (Setabran, Iran), magnesite (Birjand, Iran), alumina (Alteo pfr20, France), and titania (Cotiox KA100, Korea) powders were used as the raw materials (Table 1). This composition was chosen in this study according to the Corning Code 9606 which was considered a typical commercial cordierite glass-ceramic (SiO_2: 56.0, Al_2O_3: 19.7, MgO: 14.7, TiO_2: 9.0, CaO: 0.11, and As_2O_3: 0.5 wt%). The excess of SiO_2 and TiO_2 compared to stoichiometric cordierite helps the nucleation of crystals as the nucleating agents [6, 12]. The main reason for selecting this composition is that it provides low dielectric losses, good strength, and also thermal shock resistance. The raw materials were jar milled for 24 h using alumina balls and water. The mixed powders were dried and then melted in a furnace at the temperature of 1580 °C for 2 h. The melted materials were quenched in cold water to prepare frit. The frit was crushed and milled again in water for 48 h. Fig. 1 shows the scanning electron microscopy (SEM) and particle size distribution (PSA) of the prepared frit. Wide particle size distribution was observed for the prepared frit (0.4–30 μm) with a d_{50} of about 3.16 μm. 0.5 wt% of Dolapix PC75 was used as a dispersant. The solid volume was 70 wt%. The prepared suspension was cast in a mold and then the green bodies were dried at the temperature of 110 °C for 12 h.

The glass powder was sintered in three ways: one-step, two-step, and three-step sintering (Fig. 2). The mentioned temperatures and holding times were selected according to the other studies. Differential thermal analysis (DTA, PL-STA 1640, England), Dilatometry (DLI-402E, Germany), X-ray diffraction (XRD, Siemens D500), 3-point mechanical strength (2×3×5 mm^3, rate of 1 mm/min), and SEM (S360 Cambridge) were used to analyze the samples. Density and porosity were measured according to ASTM C20-2015. The dielectric constant of samples was measured by an LCR meter (8110G, Taiwan). At least three samples were used for each test.

Table 1. Chemical analysis of the raw materials (wt%).

Oxides	Silica	Alumina	Magnesite	Titania
SiO_2	99.34	< 0.10	< 1.00	-
Al_2O_3	0.23	99.50	< 0.50	-
MgO	< 0.10	-	46.90	-
TiO_2	-	-	-	> 98.00
Fe_2O_3	< 0.04	< 0.15	-	-
Na_2O	< 0.01	< 0.05	-	-
CaO	< 0.03	< 0.02	-	-
L.O.I.	0.28	0.20	52.00	0.40
wt% in batch	**56.20**	**19.80**	**30.87**	**8.90**

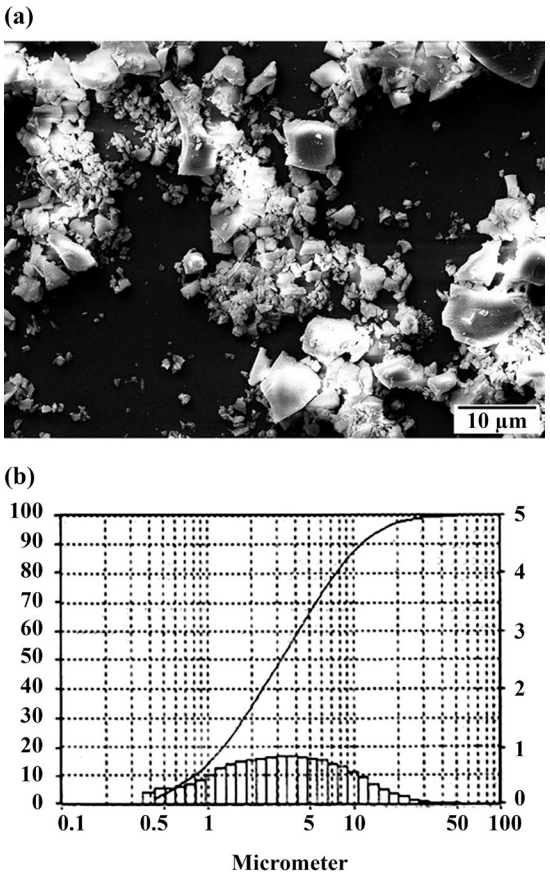

Fig. 1. a) SEM micrograph and b) PSA of the glass powder.

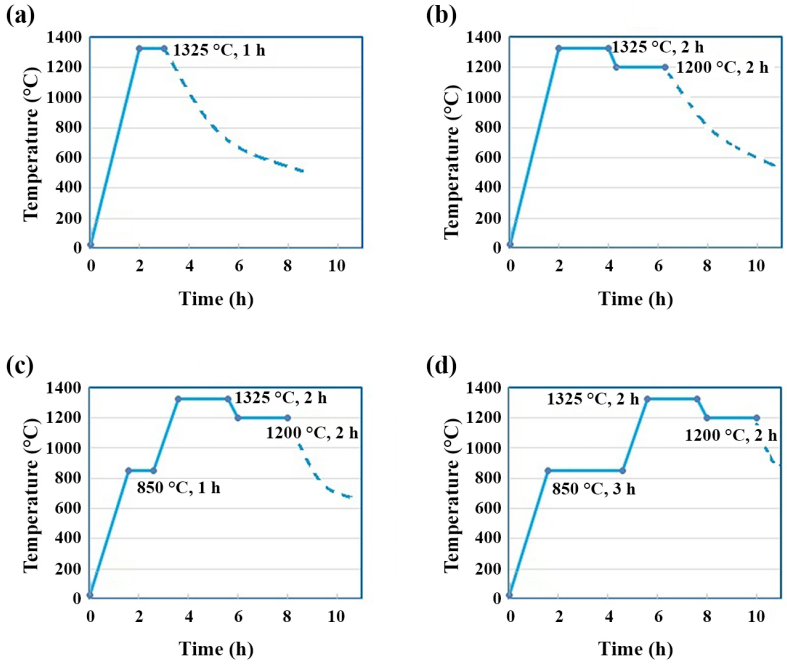

Fig. 2. Heat treatment schedules of glass powders: a) one-step, b) two-step, and c, d) three-step sintering.

3. Results and discussion

It is well-known that the densification of glass powders occurs through a viscous flow mechanism in which the molten glass phase flows between the glass particles and fills the remained porosities [13]. Since the main mass transition takes place above the dilatometric softening point temperature (T_d), densification through the viscous flow mechanism starts above this indicative temperature and takes up to liquidus temperature [14–16]. In the case of glasses with crystallization tendency, there is a competition between densification and crystallization, so high crystallization rates disrupt densification. In this state, the main portion of densification occurs in the temperature interval between the dilatometric softening point and crystallization temperatures.

3.1. Crystallization behavior of the starting glass

Fig. 3 shows the DTA thermograph of the starting glass powder at the heating rate of 10 °C/min. Based on this figure, the dilatometric softening point temperature is located at 820 °C, whilst three exothermic peaks are detectable at 900, 930, and 1177 °C which can be assigned to the crystallization effects [1]. As previously confirmed in other research [2, 3], cordierite starts to crystallize at about 900 °C in the glass matrix showing a broad exothermic peak in the relevant DTA thermograph. In addition, glass transition temperature (T_g) is located at about 765 °C, based on this figure.

In order to identify the crystalline phases developed at each crystallization peak temperature, glass powders were separately heat treated at each crystallization peak temperature for 15 min at the heating rate of 10 °C/min. The heat-treated glasses were then subjected to the XRD analysis. Fig. 4 depicts the XRD patterns of heat-treated specimens. It is evident from Fig. 4 that beta quartz solid solution precipitates as the single crystalline phase at 900 and 930 °C. By increase of temperature up to 1177 °C, cordierite is observable as the main crystalline phase while beta quartz solid solution, titanium oxide, and magnesium titanate are present as the minor crystalline phases.

3.2. Phase development and sinterability of glass-ceramics fabricated through one-step sintering

To evaluate the sinterability of starting glass powder, bulk density, water absorption, and volume shrinkage were measured at the temperature interval of 800–1325 °C. Fig. 5 shows the variations of sinterability parameters versus one-step sintering temperature.

The shrinkage profiles of cordierite glass powder indicate a shrinkage during the early stage of sintering which follows by an expansion; the expansion was related to the pore formation during crystallization [17].

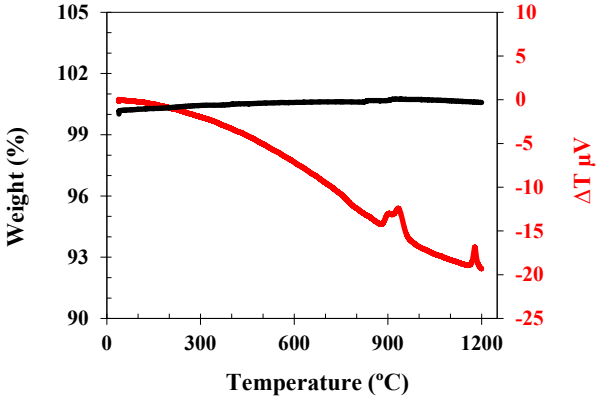

Fig. 3. DTA thermograph of the starting glass powder at the heating rate of 10 °C/min.

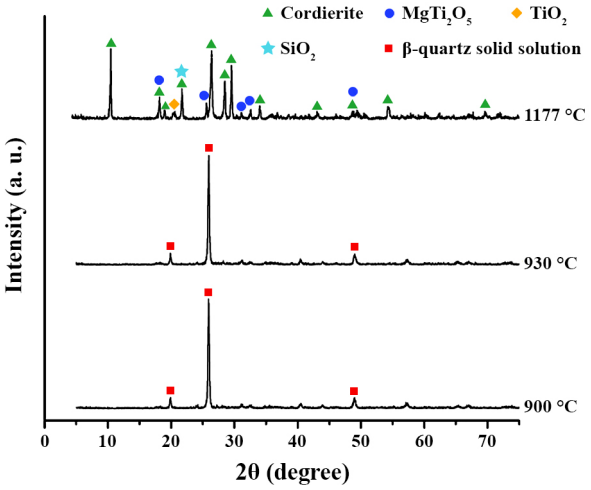

Fig. 4. XRD patterns of the glass powders heat treated at 900, 930, and 1177 °C for 15 min.

From Fig. 5, four separate temperature ranges were chosen to deeply explore the simultaneous sinter-crystallization behavior.

Stage 1: 800–900 °C

Since the selected temperature range is higher than the glass transition temperature of the examined glass (765 °C), densification occurs

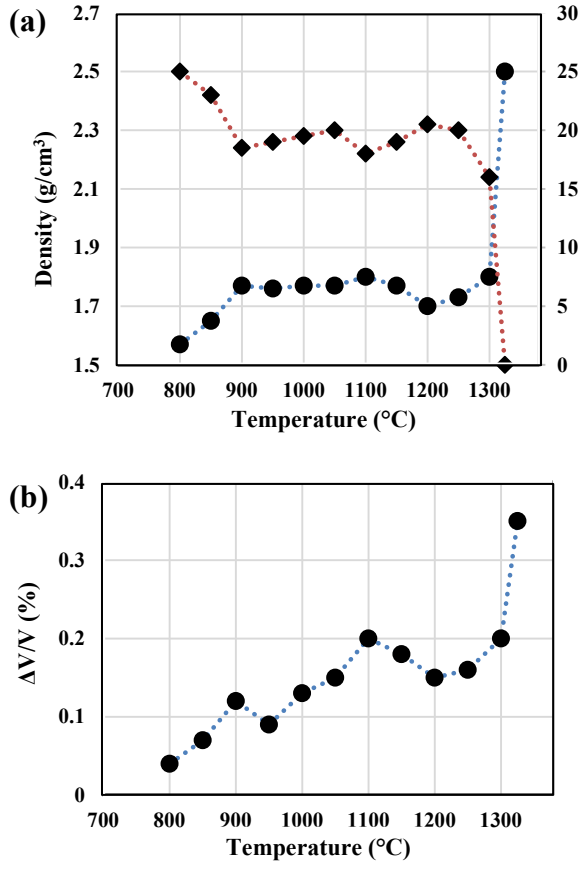

Fig. 5. Sinterability parameters versus one-step sintering temperature, a) density and water adsorption and b) shrinkage.

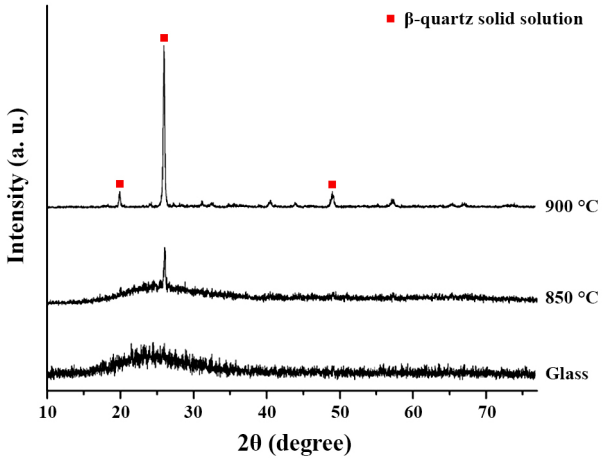

Fig. 6. XRD patterns of the glass powder and the samples heat treated at 850 and 900 °C for 1 h.

through a viscous flow mechanism. In this regard, bulk density and volume shrinkage increase by increasing temperature in this range; whilst water absorption follows a decreasing trend. Fig. 6 shows the XRD patterns of the starting glass powder along with the powder heat treated at 850 and 900 °C. Obviously, there is no sign of an undesired crystallization effect in the as-prepared glass. The small peak of β-quartz solid solution is detectable after heat treatment at 850 °C. By increasing the temperature up to 900 °C, the peak lines of β-quartz solid solution have been intensified. Since the selected temperature interval is higher enough than the glass transition temperature (T_g) densification slightly occurs with an increase in temperature. However, effective crystallization of the β-quartz solid solution interrupts densification so that sinterability parameters are not satisfying at the end of this step.

Stage 2: 900–1100 °C

At this stage, bulk density has been mildly increased by the increase of

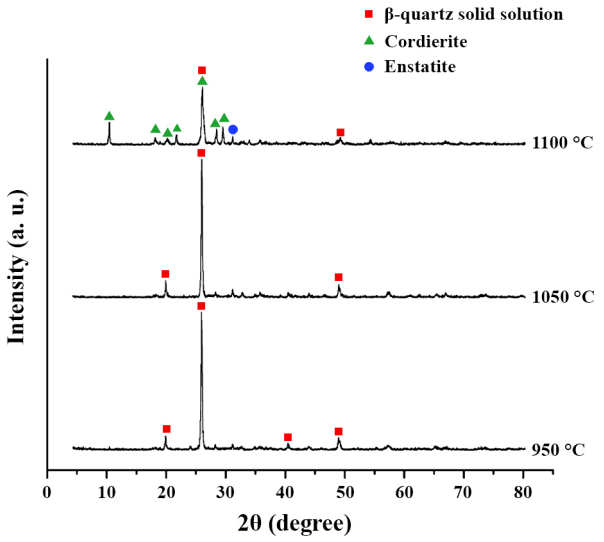

Fig. 7. XRD patterns of the samples heat treated at 950, 1050, and 1100 °C for 1 h.

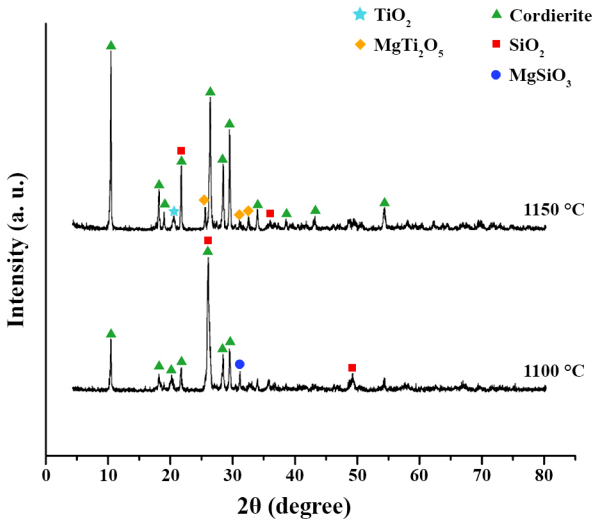

Fig. 8. XRD patterns of the samples heat treated at 1100 and 1150 °C for 1 h.

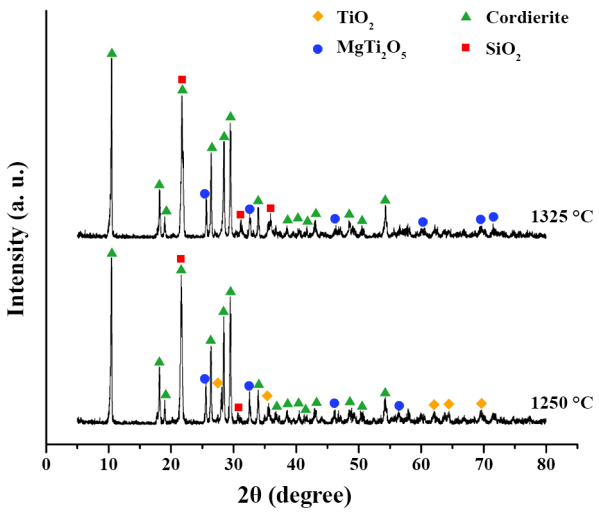

Fig. 9. XRD patterns of the samples heat treated at 1250 and 1325 °C for 1 h.

temperature without any significant changes; whilst volume shrinkage has followed a sharp increasing trend meaning occurrence of densification. In this temperature interval, water absorption has been increased by the heat treatment temperature. Fig. 7 shows the phase evolution during this stage. On the basis of Fig. 7, as the temperature increases to 1100 °C, cordierite is detectable and β-quartz solids solution has totally disappeared at this temperature. The crystallization temperature of cordierite in the MAS system was reported to be in the range of 1000–1100 °C which can lower with the impurities [14, 15]. Also, a minor amount of enstatite ($MgSiO_3$) phase can be observed.

Stage 3: 1100–1200 °C

At this stage, bulk density and volume shrinkage were decreased and water adsorption was increased with increase in temperature. It is evident from Fig. 8 that effective crystallization of cordierite occurs at 1150 °C. This temperature is in accordance with the second crystallization peak of the DTA thermograph (see Fig. 3). Additionally, magnesium titanate ($MgTi_2O_5$) is also detectable as the minor crystalline phase precipitated at 1150 °C. Presumably, a considerable difference between the density of magnesium titanate and the density of the remained glass phase is responsible for the suppressed sinterability at this temperature interval. The transformation of the amorphous phase with high density into lower density cordierite can also be responsible for the expansion [17]. However, it is worth mentioning that cordierite and the starting glass have similar bulk density values.

Stage 4: 1200–1325 °C

According to Fig. 9, the main part of the densification process occurs at this stage. At the end of this temperature interval, volume shrinkage as well as bulk density reach the maximum values; keeping water absorption equal to zero. Undoubtedly, decreased viscosity of the remained glass phase is responsible for improved densification in this stage. As shown in Fig. 9, cordierite along with magnesium titanate are still the predominant crystalline phases. Therefore, it can be concluded that 1325 °C is the optimized temperature of the one-step sintering process. It is worth mentioning that further increase of heat treatment temperature up to 1350 °C led to significant deformation of heat-treated specimens as a result of too lowered viscosity of glassy phase.

Fig. 10 shows the SEM micrographs and the EDS spectra taken from the chemically etched glass-ceramic specimens sintered at 1325 °C. In the SEM micrographs, the interconnected grey phase shows the remained residual glass. According to this micrograph, the glass phase has filled the porosities, adequately and there is no sign of considerable amounts of pores.

It should be noted that the triple eutectic point in the phase diagram of the ternary MgO-Al_2O_3-SiO_2 system is located around 1355 °C. It has been proposed that the addition of 9 wt% of titanium oxide shifts the eutectic temperature to 1330 °C [18]. Therefore, it seems the chemical composition of the studied glass lies near the eutectic temperature. As a result, melting and forming the relevant glass is relatively easy. Furthermore, this composition offers a lower crystallization tendency compared to the stoichiometric composition of cordierite [18, 19]. Suppressed crystallization tendency in turn guarantees acceptable densification behavior during sintering heat treatment owing to the diminished interference between crystallization and densification steps. The formation of minor crystalline phases such as magnesium titanate is inevitable because liquid-liquid phase separation takes place prior to crystallization. In fact, when the glass separates into two different glassy phases the chemical composition is not the same in the phase-separated areas. So, minor crystalline phases start to precipitate in these areas because of the increased concentration of their constituents.

On the basis of the EDS spectra (see Fig. 10b), chemical compositions of grey and white areas are corresponded to those of starting glass and cordierite, respectively.

Since densification of cordierite powder through the solid-state method is somehow complicated due to the narrow sintering temperature range of cordierite, fabrication of cordierite glass-ceramics via concurrent sinter-crystallization method seems a reasonable replacement [20, 21]. It has been proposed that the fierce competition between densification and crystallization of cordierite could result in complete densification using very small primary glass particles and the occurrence of densification before crystallization.

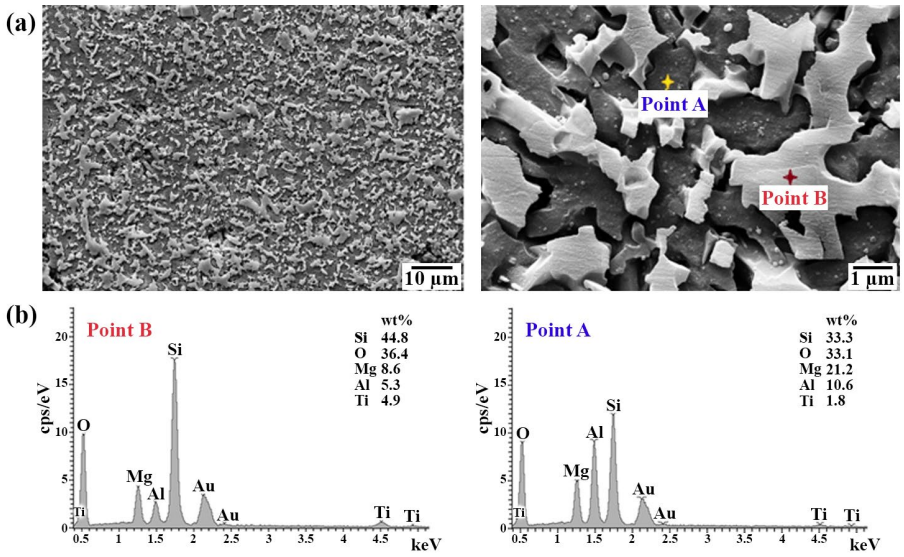

Fig. 10. a) SEM micrographs and b) the EDS spectra taken from the specimen heat-treated at 1325 °C for 1 h.

3.3. Multi-step sintering procedures

Through a one-step sintering procedure, it was found that the initiation temperature of the crystallization step is about 850 °C and the initiation temperature of the cordierite formation is about 1100 °C. According to these results, the three-step sintering temperatures were selected. In order to achieve appropriate densification, the sintering process should be completed before crystallization. The glass-ceramics with desired properties can be prepared by control of two main nucleation and crystal growth processes. Therefore, different sintering conditions (two- and three-step sintering procedures) were also carried out to complete crystallization and also improve densification behavior. Fig. 11 shows the SEM micrographs of the specimens heat-treated through multi-step sintering procedures.

In order to compare the properties of the prepared samples in this study through slip casting, one other sample was also prepared through melt casting and consequent heat treatment (as a routine procedure of the glass-ceramic preparation) and named the "bulk sample". The bulk sample was densified by two-step sintering. Here, the surfaces of the glass particles are the nucleation sites for crystallization. According to Fig. 11, the formation of well-shaped, long acicular cordierite crystals within the glassy matrix is evident. The small cordierite crystals are uniformly distributed throughout the matrix. A large number of small crystals is the desired microstructure of glass-ceramics [1]. The high aspect ratios of some crystals are also remarkable (Fig. 11c). The formation of more crystal seeds shows that the glass was kept for a longer time at the sintering temperature. The interlocked cordierite crystals embedded in the glassy matrix are also noticeable in the specimens. These microstructures show that the nucleation and crystal growth process were well controlled. Pores are barely discernible in the samples, indicating complete densification.

3.4. Measurements of physical and mechanical properties

Table 2 shows the mean values of bulk density and water adsorption of densified samples after multi-step sintering heat treatments. It can be observed that the density was decreased by increasing the crystallization. This fact is more obvious in the d sample (2.43 g/cm^3) which is indicating that the crystallization overcomes the sintering.

One of the most important features of cordierite glass-ceramics is their resistance to climatic effects. To achieve this, the porosity should be zero in the material, which provides its resistance to high moisture [6]. Dense cordierite glass-ceramics are necessary to achieve high mechanical strength and low dielectric loss. 3% porosity was reported in other papers even with the use of sintering aid [22]. The lowest porosity of cordierite glass-ceramic prepared through slip casting of oxide powders was about 0.2% [8]. The formation of pores is one of the key deteriorating factors that affects the dielectric properties and mechanical strength of glass-ceramics [14]. Table 2 and Fig. 11 show that the pore-free microstructures and water-resistance of the prepared samples are remarkable and the selected sintering conditions can improve the microstructure and physical properties of the samples.

The dielectric constant of cordierite glass-ceramics is one of the important factors that influences their performance and signal propagation [15]. A low dielectric constant is desired for these samples. Table 3 shows the dielectric constant, mechanical strength, and coefficient of thermal expansion (CTE) of the prepared glass-ceramic and the bulk sample. These are the main parameters that determine the application of these samples. Dielectric constants of the samples were measured at the frequencies 1–10 MHz. It can be seen that the prepared glass-ceramic sample (d code) has a lower dielectric constant at all measured frequencies compared to the bulk sample.

Typical dielectric constants of the cordierite-bearing glass-ceramics have been reported less than 5 [4]. The dielectric constant of cordierite glass-ceramic at 1 MHz was reported to be 5.7 which increased by the formation of the secondary phase (9.9) [22]. Other researchers [23] also obtained the dielectric constant of about 6–8 for the MAS system. Many factors influence the dielectric constant of the glass-ceramics such as crystalline phases, crystallinity, chemical composition of the glassy phase, and microstructure [15]. The main factor that distinguishes the lower dielectric constant of the prepared sample in this study compared to the bulk sample is its lower porosity, fine microstructure, and well-shaped crystals. Dense cordierite glass-

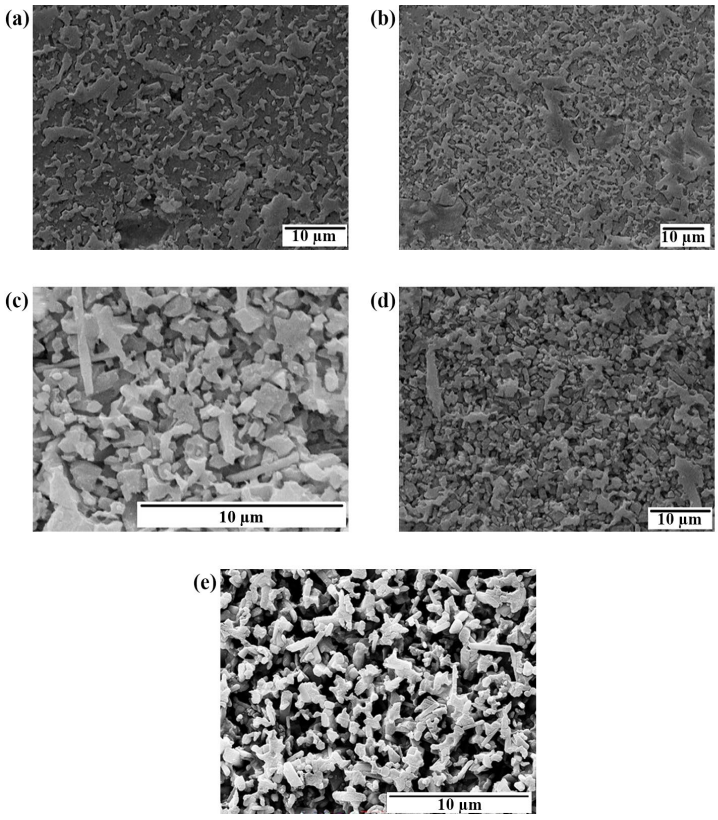

Fig. 11. SEM micrographs of the samples heat-treated through a) one-step, b) two-step, c, d) three-step sintering procedures, and e) the bulk sample.

ceramics show high mechanical strength and desired dielectric properties [14].

The mechanical strength of glass-ceramic sample (150 MPa) was also higher than that of the bulk sample (120 MPa). The prepared glass-ceramic has a higher density and crystallinity compared to the bulk sample. Therefore, improvement in the mechanical strength is expected in this sample. Also, the glass-ceramic sample has lower CTE rather than the bulk sample. Hence, it would tolerate higher thermal shock and represent higher shock resistance. The obtained CTE for the prepared sample is as low as the other cordierite glass-ceramics (3.79×10^{-6}/°C [15] and $4.4–5.6\times10^{-6}$/°C [23]).

Bending strength of lower than 100 MPa [5], 53.5 MPa [8], and 75–180 MPa [14], 110–150 MPa [15], as well as pore-containing microstructure, were reported in other studies. Comparing the results of bending strength with previously reported findings confirms that a high bending strength value was obtained in this study. High mechanical strength is required for these glass-ceramics to operate in harsh conditions and prolong their lifetime.

It is well known that the mechanical properties of glass-ceramics are greatly influenced by the porosity, degree of crystallinity, and secondary crystalline phases [8]. The porosity of the prepared samples was zero, herein; the high strength can be attributed to the absence of porosity, fine microstructure, and well-formed crystals, as well as the distribution of cordierite crystals in the glass matrix which can resist the crack propagation. Large numbers of small crystals which were distributed uniformly all over the glass-ceramic resulted in a high strength value [1]. High flexural strength obtained in the prepared samples allows them to operate more qualified in critical conditions.

Table 2. Density and water adsorption of the prepared samples (samples are coded according to Fig. 2).

Sample	a	b	c	d
Density (g/cm^3)	2.43	2.47	2.50	2.50
Water adsorption (%)	0	0	0	0

Table 3. Dielectric constant, strength, and CTE of the prepared samples.

	Frequency (MHz)	Glass sample (d code)	Bulk sample
Dielectric constant	1	5.87	7.43
	2	6.06	6.93
	3	6.16	7.17
	4	4.59	6.35
	5	5.28	7.50
	6	5.53	7.18
	7	6.39	7.88
	8	6.51	7.78
	9	8.77	10.15
	10	9.12	10.37
Strength (MPa)		150±15	120±10
CTE (10^{-6}/°C) (36–600 °C)		3.79	4.13

4. Conclusions

Sintering conditions of cordierite glass-ceramics prepared through slip casting have been investigated in the present work. First, the glass powder was obtained via the classic melt quenching technique and then the slip-casted glass powder was sintered under different sintering conditions. The density and water adsorption of the glass-ceramic which has been carried out by one-step sintering at 1325 °C for 2 h, was 2.40 g/cm^3 and 5%, respectively. Three-step sintering has resulted in higher densification (2.50 g/cm^3), lower water adsorption (0%), and finer microstructure compared to one- and two-step sintered samples. The strength and coefficient of thermal expansion of the prepared glass-ceramic through slip casting were also higher than that of the bulk sample prepared by the common melting and casting procedure as a reference sample. The results showed that slip casting has the potential of preparing cordierite-based glass-ceramic which can provide desirable advantages such as zero porosity, low thermal expansion coefficient, and dielectric constant.

CRediT authorship contribution statement

Mohammad Javad Maleki: Investigation, Writing – original draft, Formal Analysis, Software.
Hudsa Majidian: Supervision, Methodology, Project administration, Funding acquisition.
Sara Banijamali: Validation, Writing – review & editing, Data curation.
Mohammed Zakeri: Supervision, Methodology, Project administration, Funding acquisition.

Data availability

The data underlying this article will be shared on reasonable request to the corresponding author.

Declaration of competing interest

The authors declare no competing interests.

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