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Effect of Sintering Time on Crystal and Structure of Chlorine-containing Low-titanium Slag Glass-ceramics

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Abstract. Glass-ceramics with akermanite, diopside, spinel and perovskite crystalline phases were prepared by using the direct sintering method and using chlorine-containing low-titanium slag as a raw material. The changes in crystalline phase composition, relative content and microstructure of glass-ceramics sintered at different times were studied. The results suggested crystalline phases remained steady while the relative content of each phase changed significantly. The relative content of akermanite decreased firstly and then increased while those of diopside changed oppositely. The relative content of spinel rose from 6.6% to 9.3% and perovskite stabilized at 10.0-11.1%. In addition, the pore size of glass-ceramics declined firstly and then increased. The optimal microstructure of glass-ceramics were obtained when crystallized at 890 °C for 60 min and sintered at 1185 °C for 60 min. In general, glass-ceramics with compact microstructures could be obtained at appropriate sintering times.

1. Introduction

Chlorine-containing low-titanium slag (CTS) consists of low-titanium industrial waste residue produced during extraction of titanium components from Ti-bearing blast furnace slag [1]. The process typically involves two steps: high-temperature carbonization and low-temperature chlorination, with latter step producing 2% to 5% chlorine in waste residue. The chlorine-containing low-titanium slag cannot be used as building material like ordinary waste residues due to presence of chlorine considered as main culprit in erosion of reinforced concrete [2]. So far, chlorine-containing low-titanium slag can only be stored in open areas and wasting lands due to lack of reasonable utilization. However, its migration and release of chlorine cause pollution to water and soil environment. Therefore, finding novel ways for treatment of chlorine-containing low-titanium slag as industrial solid waste is highly desirable for environmental remediation.

Chlorine could be removed from chlorine-containing slag by heat treatment [3]. However, dechlorination residues obtained by this method cannot effectively be utilized. Therefore, it is necessary



to design more reasonable means for utilization of dechlorinated waste residues while removing chlorine. The preparation of glass-ceramics from waste slag is a high value-added method for effective utilization of waste slag [4-6]. At the same time, the harmful components present in waste slag can be purified in the process of preparing glass-ceramics. Compared with ceramic materials, glass-ceramics from waste slag have higher densities, lower or none water absorptions, and elevated strengths. Therefore, it can be used as building and decorative material [7]. However, high-temperature melting process as necessary step for preparation of glass-ceramics, greatly increases both production and environmental costs, therefore limiting large-scale production of glass-ceramics [5].

Some studies demonstrated that glass-ceramics could be obtained by direct sintering at low temperatures from green bodies pressed from ground mixtures of waste residues and additives [8-10]. This route is not only economical but yields glass-ceramics with properties comparable to those produced by conventional methods, such as bulk crystallization and melt-sintering. In addition, direct sintering is similar to ceramic sintering, meaning that production and preparation of glass-ceramics can achieve continuity and automation as ceramics. Moreover, existing ceramic production equipment can directly be employed to produce glass-ceramics, which is beneficial to development of glass-ceramics industry. On the other hand, sintering is indispensable no matter the used sintering method to prepare glass-ceramics. Moreover, both temperature and time are process parameters requiring strict control during sintering to determine the sintering effect of glass-ceramics. Numerous studies discussed the effects of sintering temperature on glass-ceramics. Some found that increasing sintering temperature could accelerate the sintering process and contribute to formation of dense structures with good mechanical properties in early and middle stages of sintering [8,11,12]. However, improving the properties of glass-ceramics by adjusting sintering temperatures becomes difficult in late stage of sintering. Therefore, as sintering temperature tends to be stable, adjusting the sintering time is necessary for formation of dense microstructural glass-ceramics with improved properties. Meanwhile, reports on the influence of sintering time on the crystalline phase and microstructure of chlorine-containing low-titanium Slag glass-ceramics prepared by direct sintering have not been published.

Here, feasibility of preparing glass-ceramics with good microstructures from chlorine-containing low-titanium slag using direct sintering was demonstrated. The effects of sintering time on crystalline phase composition, relative content and microstructure of glass-ceramics were all investigated. The results suggested that sintering time has a significant effect on phase content and microstructure of glass-ceramics and usage of chlorine-containing low-titanium slag as raw material in glass-ceramics preparation by direct sintering could be a good resource utilization method for chlorine-containing low-titanium slag, which is conducive to environmental protection and sustainable development.

2. Experimental

2.1. Raw materials

The chlorine-containing low-titanium slag (CTS) black granular powder was collected from Panzhihua, China. The presence of chloride in CTS induced strong moisture absorption, resulting in CTS with water content of 5.28%. Therefore, CTS should be dried before using.

2.2. Preparation of glass-ceramics

CTS was first placed in an oven at 105 ± 5 °C for 24 h, then dried CTS was ball milled in planetary ball mill at 200 r/min for 4 h. The product powder was passed through a 160 mesh sieve (<96 μm) to yield ground powder. On the other hand, the retained CTS was recycled to ensure maximum use of resources. Without adding binder, the ground powder was uniaxially pressed in steel mold at 10 MPa to yield cylindrical compacts ($\varnothing 25.3$ mm $\times$ 5.5 mm) and rectangular compacts (5 mm $\times$ 5 mm $\times$ 50 mm).

The two compacts were placed on a corundum sheet containing alumina powder and placed in an electric resistance furnace, heated from room temperature to crystallization temperature at a rate of 5 °C/min then kept at crystallization temperature for 1 h. Next, the temperature was raised from crystallization temperature to sintering temperature at a rate of 5 °C/min then kept at sintering

temperature for 30 min, 60 min, 90 min and 120 min, respectively. After cooling to room temperature, glass-ceramics with two different shapes were obtained. The glass-ceramics samples obtained at different sintering times are listed in Table 1.

Table 1. Glass-ceramics obtained at different sintering times.

Sintering time (min)	30	60	90	120
Samples	S-30	S-60	S-90	S-120

2.3. Characterization

The chemical compositions of CTS specimens were identified by X-ray fluorescence spectroscopy (XRF, Axios, PANalytical, Netherlands).

The phase structures of CTS and glass-ceramics were examined by X-ray diffraction (XRD, Ultima IV, Rigaku, Japan) using Cu K α radiation at operating conditions of 40 kV voltage, 40 mA current, 2 θ range from 10-70°, and scanning speed of 4° (2 θ) min⁻¹ at a step size of 0.02°. The crystalline phases and relative content were estimated using MDI Jade 6 software. In this study, The “Whole Pattern Fit” refinement function in MDI Jade 6 was used to calculate the quantitative analysis of the relative content of each crystalline phase in glass-ceramics. The refinement process is referred to works of Zhao et al [13]. The background curve is 5th-Order Polynomial, the profile shape function is pseudo-Voigt, and the refinement range is 2 θ =10-70°. In order to the evaluation of refinement results, R-factor value and E-factor value are considered as the criteria for judging the quality of refinement. The ratio of R/E is called the “goodness of fit”, which is approximately 1, the refinement results are better [14].

The thermal behaviors of CTS were analyzed by differential thermal analysis and thermogravimetry (DSC/TG, STA 4499F5, Netzsch, Germany) from 30 to 1200°C at constant heating rate of 10°C/min using Al₂O₃ crucible as reference material. The microstructures of glass-ceramics were observed by scanning electron microscopy (SEM, Ultra55, Carl zeissNTS GmbH, Germany).

3. Results and discussion

3.1. Characterization and thermal behaviors of CTS specimens

The phase and chemical compositions of CTS specimens are gathered in Fig. 1 and Table 2, respectively. In Fig. 1, CTS showed amorphous phase with crystalline phases of khamrabaevite (PDF#71-0298) and carbon (PDF#75-0444). At around 26° is a broad peak which belongs to 002 peak of carbon crystallite of coke and the value of d₀₀₂ is 3.42Å. Coke is an important raw material for high-temperature carbonization process of Ti-bearing blast furnace slag, which can promote the formation of khamrabaevite. In general, some of amorphous coke are converted to structurally regular carbon crystallite at high temperatures [15]. The carbon crystallite 002 peak of coke reflects the regularity of the carbon structure, a weak and broad 002 carbon peak in CTS indicates low ordering of carbon structure [16]. Khamrabaevite is formed during high-temperature carbonization and remained in CTS due to incomplete chlorination at low temperature [17]. The main chemical components of slag were identified as CaO, SiO₂ and Al₂O₃, with certain amounts of TiO₂, Fe₂O₃ and MgO (Table 2). The chemical composition of CTS met the requirements for formation of glass-ceramics. Besides, TiO₂ and Fe₂O₃ could act as nucleating agents, contributing to devitrification of glass-ceramics [18]. Theoretically, CTS could be used as component during preparation of glass-ceramics. Here, CTS was used as raw material for preparation of glass-ceramics, which could reduce the production cost of glass ceramics. On the other hand, this made it feasible to analyze the effects of sintering time on glass-ceramics without interfering additives.

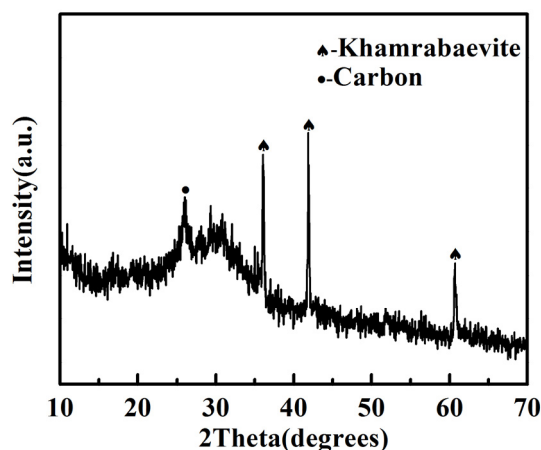


Fig. 1 XRD pattern of CTS specimens.

Table 2 The chemical compositions of CTS specimens (wt%).

CaO	SiO ₂	Al ₂ O ₃	TiO ₂	MgO	Fe ₂ O ₃	Cl	Others	Loss
28.30	24.18	11.66	7.57	6.65	3.83	2.58	2.45	12.78

The DSC-TG curves of CTS specimens are shown in Fig. 2. The endothermic valley observed in DSC at 70 °C was related to loss of adsorbed water molecules on CTS. As temperature increased, the exothermic peak at 890 °C became prominent. This peak could be used as crystallization temperature of glass ceramics. The TG curve depicted continuous weight loss. The weight loss below 140 °C was mainly caused by evaporation of moisture and that of 5.6% recorded between 140 °C and 1130 °C was caused by volatilization of carbon dioxide and chlorine-containing gas [3]. Carbon dioxide was formed by oxidation of carbon and khamrabaevite. At temperatures higher than melting point, chloride would be converted from solid state to gas and escape from CTS, releasing chlorine-containing gas. The TG curve between 1130 °C and 1200 °C showed 1.4% weight loss caused by release of SO₂ from alkali metal sulfates at high temperatures [19]. Meanwhile, the endothermic trends in DSC curve were attributed to melting of low melting point components.

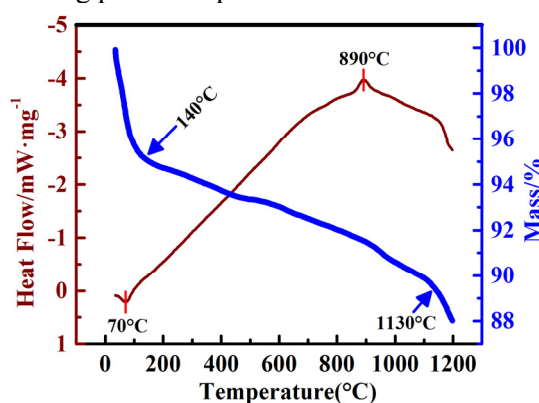


Fig. 2 DSC-TG curves of CTS specimens.

3.2. Heating treatment of glass-ceramics

Using conventional melt-sintering of glass-ceramics, the sintering process would require completion before crystallization. Besides, reduction in compactness caused by rapid growth of crystals during sintering should be prevented [20]. In direct sintering, the sintering process was performed simultaneously during or after crystallization [21]. In direct sintering of glass-ceramics using

water-quenched slag as raw material, the sintering temperature was much higher than crystallization temperature [22]. This would result in two independent temperature ranges related to sintering and crystallization. Therefore, crystals could grow at crystallization temperature while green compacts might densify at sintering temperature. This would prevent negative influence of crystal growth on compactness of green compacts. For the above reasons, the sintering temperature and crystallization temperature were set separately. Therefore, the preparation of glass-ceramics from CTS by direct sintering required heat treatment process to be set in two steps: crystallization and sintering. Combined with preliminary experiments, the best complete heat treatment process can be determined, consisting of first heating from room temperature to crystallization temperature (890 °C) at 10 °C/min and then kept at this temperature for 60 min to allow crystallization to occur. This was followed by further heating from crystallization temperature (890 °C) to sintering temperature (1185 °C) and then kept at this temperature for 30 min, 60 min, 90 min, and 120 min. Finally, the obtained product was naturally cooled to room temperature.

3.3. Crystalline phase composition and content of glass-ceramics

The XRD patterns of glass-ceramics prepared at different sintering times are gathered in Fig. 3. The crystalline phases of all specimens consisted of akermanite (PDF#79-2424), diopside (PDF#87-2070), spinel (PDF#75-1799) and perovskite (PDF#86-1393). This indicated that changes in sintering time had no influence on crystal phase composition of glass-ceramics. The differences observed in diffraction peak intensity of each crystalline phase of glass-ceramics suggested that sintering time did affect content of each crystal phase. In order to determine what sintering times did affect the content of each crystalline phase in glass-ceramics, XRD patterns of all specimens were refined.

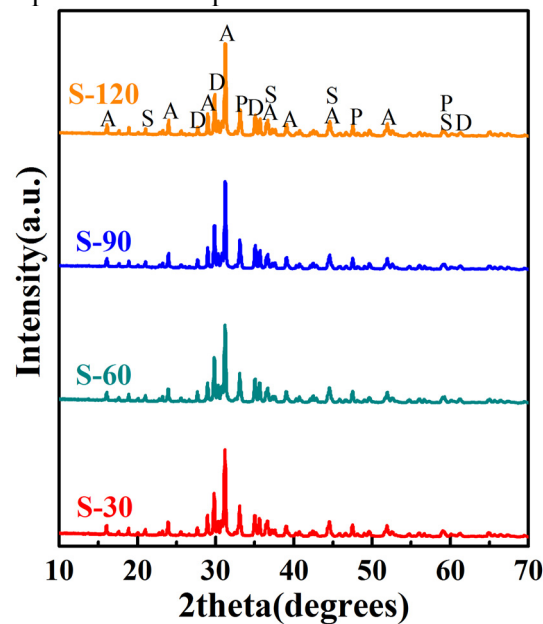


Fig. 3 XRD patterns of glass-ceramics sintered at different times: S-120 for 120min, S-90 for 90min, S-60 for 60min, and S-30 for 30min; A:Akermanite, S:Spinel, D:Diopside, P:Perovskite

Table. 3 provides the refinement results including the relative contents of crystalline phases, R-factor value, E-factor value and ratios of R/E. Ratios of R/E in all specimens are between 1.40 and 1.93, which indicates that refinement results are reliable [14]. Sintering time greatly influenced the contents of akermanite, diopside and spinel while showed little influence on perovskite. With the increase of sintering time, the relative contents of akermanite decreased firstly and then increased, but the relative contents of diopside oppositely changed. The relative content of diopside in the S-60 specimen was the highest (41.7%), while the lowest content of akermanite (40.6%). The change in relative content of crystalline phase indicated growth or shrinkage of crystal particles. Akermanite and diopside were

susceptible to faster ion migration at high temperatures, degrading stability of crystal structure. Under these circumstances, the relative ratio of bridged oxygen to non - bridged oxygen decreased due to alkali metal ions, such as Ca^{2+} . Moreover, the tetrahedral group $[\text{SiO}_3]^{4-}$ of diopside was destroyed and converted into ditetrahedral group $[\text{Si}_2\text{O}_7]^{6-}$ while octahedral magnesium group $[\text{MgO}_6]^{10-}$ changed into tetrahedral group $[\text{MgO}_4]^{6-}$ [23]. These changes promoted secondary growth of akermanite grains, decreasing diopside content and increasing akermanite content in glass-ceramics. The relative contents of spinel rose from 6.6% to 9.3%, which means the increased sintering time helped the growth of spinel crystals. The relative content of perovskite remained unaffected by sintering time and kept stable at 10.0-11.1%.

Table. 3 The relative contents of crystalline phase of glass-ceramics sintered at different times.

Samples	Phases and Contents(%)				R-factor(%)	E-factor(%)	R/E ratio
	Akermanite	Diopside Fe-bearing	Pervoskite	Spinel			
S-30	46.7(1.2)	35.7(2.4)	11.1(0.3)	6.6(1.0)	8.12	4.19	1.93
S-60	40.6(1.1)	41.7(2.7)	10.5(0.3)	7.2(1.0)	6.43	4.26	1.51
S-90	41.7(1.1)	39.2(2.5)	10.6(0.3)	8.5(0.9)	5.99	4.25	1.41
S-120	44.0(1.1)	36.7(2.4)	10.0(0.3)	9.3(1.1)	6.04	4.31	1.40

3.4. Morphological features of glass-ceramics

Fig. 4 displays the apparent morphology and microstructure of glass-ceramics obtained at different sintering times. Figs. 4(a-d) represent the surface images of S-30, S-60, S-90 and S-120, respectively. In Fig. 4(a), numerous voids appeared in glass-ceramics with loose structure. In contrast, no voids were observed in Fig. 4(b) but few tiny pores in isolated distributions appeared. The pore size dropped observably and structure looked relatively dense, indicating an increase in the compaction of the glass ceramic at a sintering time from 30 min to 60 min. As sintering time continued increasing, some isolated tiny pores present on the surface became interconnected and changed into large-aperture pores (Fig. 4(c and d)). During sintering at high temperature, solid particles of glass-ceramics would bond to each other, and voids would gradually be eliminated. Meanwhile, some liquid would flow among particles, filling the tiny pores and increasing density of glass ceramics. These macroscopic changes rose linear shrinkage rate and bulk density. Fig. 4(a) and 4(b) confirmed the microscopic changes in structure from loose to dense, suggesting S-60 with lower water absorption than S-30. Moreover, XRD indicated formation of secondary grown akermanite grains as sintering time prolonged. This would destroy the equilibrium and stable structure among liquid phase, pores and crystalline phases, exposing some closed pores and forming large pores with adverse effects on properties of both S-90 and S-120 specimens [24].

Figs. 4(e-h) shows the internal images of glass-ceramic specimens. In Fig. 4(e), grains in the forms of plate-like, columnar and granular could be clearly observed. Small-sized grains surrounded large-sized grains, or precipitated on their surface defects. With the increase of sintering time, some grains were softened and deformed, which made the crystal boundaries blurred and reduced the gaps between grains. Plate-like and columnar grains appeared to be more easily deformed, while granular grains could still maintain relatively complete structures. The deformation of crystal grains were more serious in Fig. 4(g). These deformed grains filled the interstices of particles like the amorphous "liquid phase". In Fig. 4(h), the complete columnar and plate-like grains were difficultly observed. Granular grains and amorphous "liquid phase" constituted a chimeric structure. Some studies had proven that the viscous flow of glass phase contributed to the dense sintering of glass-ceramics [25]. However, if the liquid phase was formed by soften and deformed crystal grains, the stability of the microstructure of glass-ceramics would be decreased. As a result, the sintered compactness of glass-ceramics became

deteriorated, and porosity defects would be formed on the surface of glass-ceramics. This phenomenon was confirmed in the apparent morphology of both S-90 and S-120 specimens (Fig. 4(c and d)).

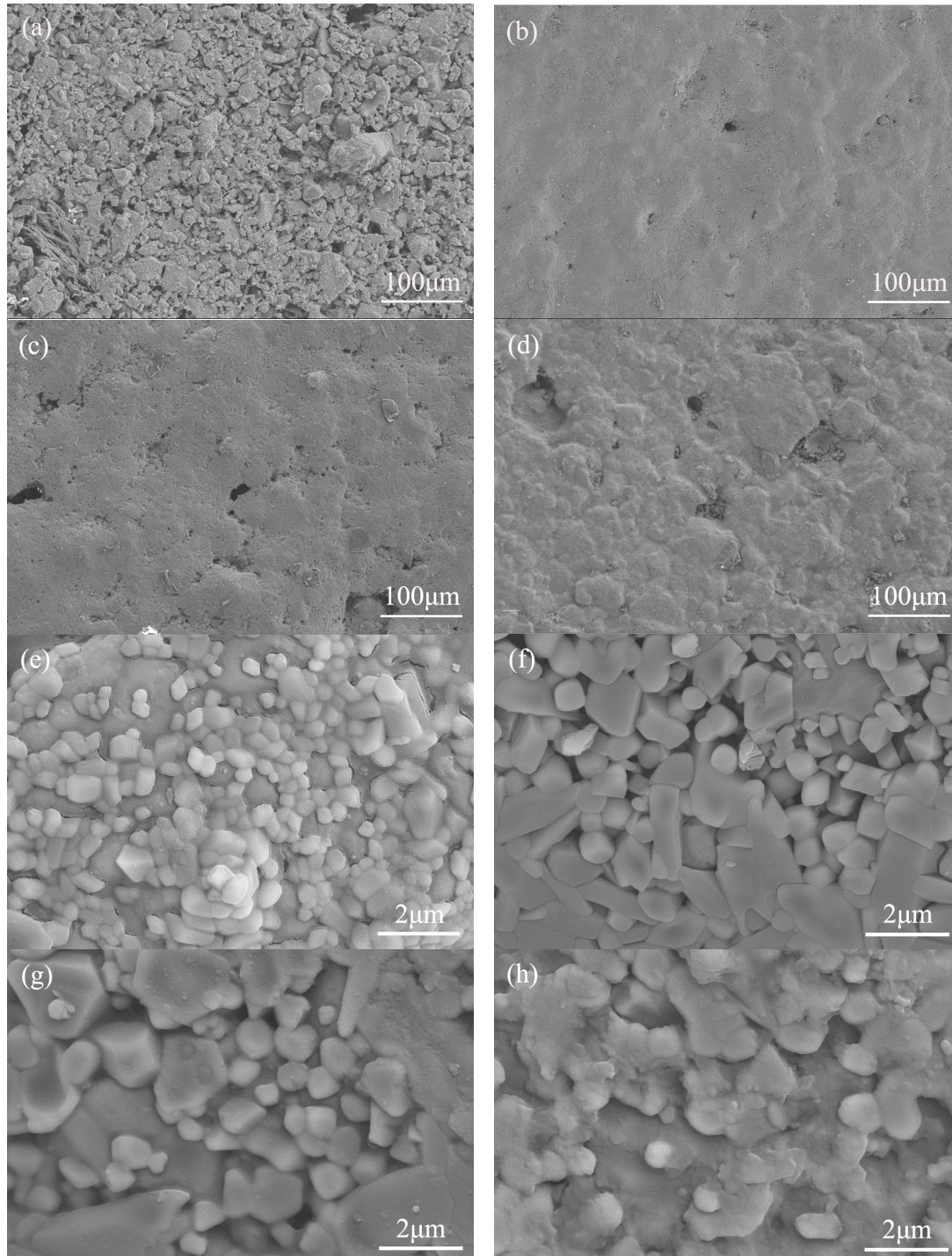


Fig. 4 Morphology of glass-ceramics sintered at different times: (a) and (e) for S-30, (b) and (f) for S-60, (c) and (g) for S-90, (d) and (h) for S-120

4. Conclusions

Glass-ceramics with akermanite, diopside, spinel and perovskite crystalline phases were prepared from chlorinate-containing and low titanium-bearing slag as raw material using direct sintering method. With

the increase of sintering time, the crystalline phase composition remained unchanged while the relative content of each crystalline phase varied. In particular, the relative content of akermanite decreased firstly and then increased, while diopside was the opposite. The relative content of spinel rose from 6.6% to 9.3% and the content of perovskite remained at 10.0-11.1%. The change in sintering time affected the microstructure of glass-ceramics, especially pore size and pore state. With a sintering time of 60 minutes, few tiny pores distributed on glass-ceramics surfaces were formed suggesting a high compaction degree of glass-ceramics. However, excessively long sintering times resulted in the size of the connected holes. These connected pores adversely affected the properties of the obtained glass-ceramics. The optimal microstructure and high diopside content of glass-ceramics were obtained when crystallized at 890 °C for 60 min and sintered at 1185 °C for 60 min. In general, the proposed method provided new way for the resource utilization of chlorine-containing low-titanium slag, as well as theoretical and technical reference for direct sintering of glass-ceramics from industrial waste.

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