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STUDY ON A NEW PROCESS AND ITS KINETICS OF IRON RECOVERY AND GLASS-CERAMICS PREPARATION FROM DESULFURIZATION SLAG

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Abstract

This paper creatively proposes a new process with desulfurization slag leached by ammonium chloride as pretreatment, and the main point of this paper lies in the processing of desulfurization residue leached by ammonium chloride. Through component analysis the formula is adjusted with high aluminum coal ash and glass cullet, making the melting point of the reduction slag around 1200 ℃, which facilitates the separation of iron and slag. At the same time, the reduction slag is adjusted to the target crystallization phase, so that the high temperature reduction slag after carbon thermal reduction can be used to produce glass-ceramics directly. The results show that iron recovery rate is over 99%, and diopside and nepheline glass-ceramics are produced, which shows that the new process is feasible. However, the crystal growth index is less than 3, which means that the crystallization capacity of the glass-ceramics is low, and nucleating agent is needed in the preparation of glass-ceramics.

Keywords: *Desulfurization slag; Glass-ceramics; Diopside and nepheline; Kinetics; Factsage*

1. Introduction

The accumulation of industrial waste has become a serious problem in many countries, because it does not only occupy a large amount of land, but also causes unforeseen environmental pollution, and waste reuse is an attractive disposal method since it can increase resource conservation and reduce or even eliminate disposal costs and potential pollution problems [1]. Steelmaking desulfurization slag refers to the waste slag obtained from the pre-desulfurization treatment of molten iron before it enters the converter [2]. Desulphurization slag is a typical bulk solid waste of iron and steel smelting, of which the utilization rate of metallurgical slag is less than 30%, and the rest is simply stacked in the slag field, wherein the total quantity has surpassed 100 million tons [3]. The main working principle is to add the desulfurizer composed of lime, limestone, and fluorite into the molten iron with high sulfur content, and make the desulfurizer and molten iron fully react through mechanical stirring to generate desulfurization products to achieve the purpose of desulfurization. Finally, the desulfurization products are extracted to obtain molten iron with low sulfur content and desulfurization slag. At present, the recycling of desulfurization slag is lacking due attention, the main treatment method is only the simple use of electromagnetic crane for bulk magnetic separation as steel scrap recycling, the rest of which returns to sintering, or takeaway for cement raw materials, road filling materials, etc. However, the bulk magnetic separation materials often bonded together as slag and iron that are directly added into the converter will inevitably cause re-sulfurization, having a bad impact to the subsequent metallurgy process and production costs. Also, after magnetic separation small iron and slag particles are entangled and cannot be separated effectively. As the grade of iron is low and fluctuates greatly, it does not only reduce the sintering grade, but also has an adverse effect on the sintering strength. It is also not economical to take away the material directly [4].

Compared with traditional ceramics and glass, glass-ceramics have many outstanding characteristics, including high mechanical strength, thermal shock resistance, wear resistance, chemical corrosion resistance and so on [5, 6]. In recent years, many scholars have prepared glass-ceramics from different types of solid wastes [7], such as iron and steel smelting waste residue [6, 8, 9], fly ash [10-12], and waste glass [13]. The composition of these solid wastes is very complex, containing many kinds of oxides, and various oxides will affect the performance of glass-ceramics. Among them, SiO_2, Al_2O_3, CaO, and MgO are the most common and have the greatest

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influence on material properties. Acidic oxide SiO_2 is essential as the forming body of the silicate network, while CaO, MgO and other oxides contribute to the phase separation and crystallization of the basic glass. Al_2O_3 is an amphoteric oxide that can form a tetrahedral glass frame [6, 8]. In addition, metallurgical solid waste may also contain TiO_2, CaF_2, and Cr_2O_3, which are conducive to nucleation, and it is conducive to reducing the addition of external nucleating agents. However, as the high content of iron in desulfurization slag, it is often difficult to prepare glass-ceramics directly, or a lot of texturizine agent should be added. Therefore, the recovery of valuable metal iron through carbothermal reduction reaction can not only be directly recycled as raw materials for iron and steel smelting, but also eliminate its influence on glass-ceramics.

Besides containing iron, desulfurization slag also contains a large amount of SiO_2, Al_2O_3, CaO, and MgO, which can be used as raw material for CaO-MgO-Al_2O_3-SiO_2 glass-ceramics (CMAS); however, desulfurization slag contains large amounts of calcium and its basicity is exorbitant in order to meet the requirements of molten iron desulfurization. Therefore, to meet the requirements of recovering metal iron and preparing CMAS glass-ceramics, it is needed to add a large amount of silica to adjust the composition of desulfurization slag, or selectively reduce calcium in slag. As the most of the calcium in the desulfurization slag exists in the form of calcium oxide and silicate, calcium can be leached by ammonium chloride solution with high selectivity[14, 15], and it will reduce the addition of texturizing agent effectively. In addition, our preliminary experiments [16, 17] also showed that the filtrate of slag leached by NH_4Cl is a high purity system of $CaCl_2$-NH_4Cl-NH_3-H_2O, and high purity and commercial calcium carbonate can be prepared by blowing carbon dioxide gas. The carbonization process was spontaneous, and ammonium chloride could be recycled during the leaching-carbonization process. Therefore, the ammonium leaching of desulfurization slag can not only reduce the calcium content in the desulfurization slag, making it more suitable for recovering metal iron and preparing CMAS glass-ceramics, but also the filtrate can sequestrate a large amount of carbon dioxide produced by steel plants, and prepare commercial calcium carbonate at a low cost. Based on this, a new process for preparing commercial calcium carbonate, glass-ceramics, and recovering iron from desulfurization slag with ammonium leaching is proposed as shown in Figure 1. In addition, desulphurization slag is produced in the steel plant, so the on-site treatment can save a large amount of transportation costs, and directly apply the existing high temperature equipment, to sensible heat of high temperature slag.

Desulfurization residue as the main raw material, which is pretreated by ammonium chloride leaching to obtain desulfurization residue leached by ammonium chloride (DRLAC) and filtrate. The filtrate is a typical $CaCl_2$-NH_4Cl-NH_3-H_2O system fed with carbon dioxide to prepare calcium carbonate has been studied in the early stage. The main point of this paper lies in the processing of DRLAC. Through component analysis, the formula will be adjusted with high aluminum coal ash and glass cullet to make the melting point of the reduction slag around 1200 °C, which facilitates the separation of iron and slag; at the same time, the reduction slag after iron recovery has been adjusted to the target crystallization phase, so that the high temperature reduction slag can be used to produce glass-ceramics directly.

2. Experimental

2.1. Material

In this study, desulfurization slag is KR desulfurizing slag without any processing as shown in Fig.2 comes from Wuhan iron and steel group. It can be seen in Table 1 that there is a lot of chemical element iron mixed in the slag, which is difficult to separate, and the calcium content in desulfurization slag is as high as 42.33% while the silicon content is

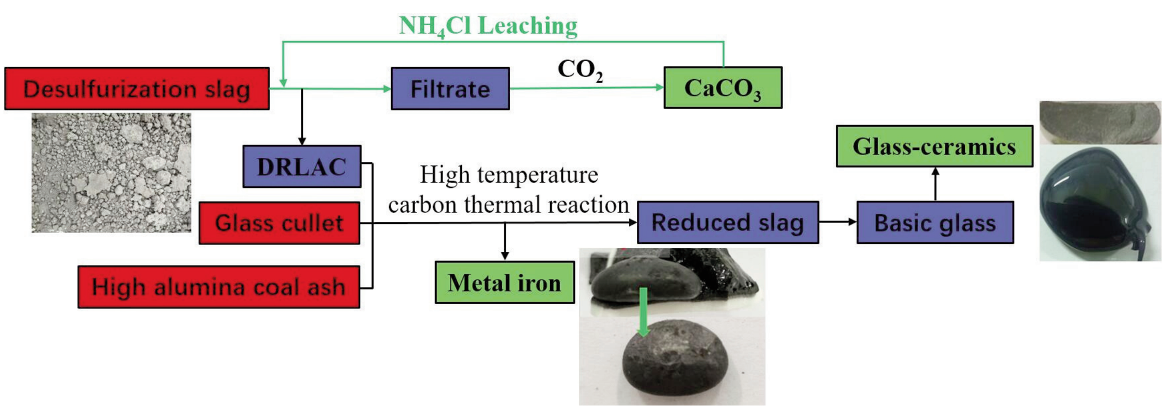

Figure 1. *A new process for preparing glass-ceramics after recovering iron from desulfurization slag*

only 6.2%, in other words, the binary alkalinity is as high as 6.83. In addition, the slag also contains aluminum, this is related to the fact that the slag on the surface of molten iron was not completely cleaned during the pretreatment of molten iron. The desulfurization slag also contains certain magnesium, titanium and sulfur.

The pretreatment of desulfurization slag is to grind the desulfurization slag to less than 60 mesh, then leach it with 4 mol/L ammonium chloride solution for 24 h according to the ratio of steel slag mass to solution volume 1:10 (g/mL). After filtration, the filter slag is washed with ultra-pure water for 3 times. After drying, desulfurization residue leached by ammonium chloride (DRLAC) is obtained. High alumina coal ash comes from a factory in Inner Mongolia, China, and its composition is shown in Table 1 below. The glass cullet is ordinary glass on the market, ammonium chloride is analytical reagent and the water used is ultra-pure water.

2.2. Preparation process

The mixed raw materials of desulfurization residue leached by ammonium chloride, high alumina coal ash and glass cullet were put into a corundum crucible and heated in a muffle furnace to conduct iron recovery experiments, in which the temperature was heated to 1500 ℃ and holding for 2 h, then the samples were cooling to room temperature within muffle furnace. After that, the crucible was taken out and opened to take macroscopic photos, weight metal iron after slag-iron separation, and analyze the iron content and phase in the reduced slag.

The reducing slag after carbothermal reduction separation was put into the corundum crucible again and heated to 1500 °C for 2 h in a muffle furnace to simulate the molten reduction slag after iron separation, and then the crucible was quickly taken out of the high-temperature furnace, and the molten reducing slag was poured onto a pre-heated steel plate. Subsequently, the steel plate was quickly moved into a muffle furnace (600 °C and 2 h) to remove the internal stress generated by the melt after rapid cooling at high temperature and the basic glass is prepared. A small amount of basic glass is ground into powder for comprehensive thermal analysis, and according to the differential thermal analysis, a controlled-crystallization method was used to obtain the glass-ceramics. Then the macroscopic photos and phases of glass-ceramics were test.

2.3. Characterization

The chemical compositions of raw material were analyzed by X-ray fluorescence (XRF) (XRF-1800, Shimadzu, Japan). The reduced slag and iron were removed from the crucible for weighing and chemical analysis (GB YB/T 148-2009) to calculate the iron recovery rate and the total iron in the slag according to Eq. (1) and Eq. (2), Where X_{Fe}(g) is iron recovery rate in desulfurization slag, M_{Fe}(g) is recovered iron amount, MT_{Fe}(g) is amount of total iron in desulfurization slag, and MRT_{Fe}(g) is the amount of total iron remaining in reduced slag. The phases of the slag were determined using an X-ray diffraction (6100 lab instrument from Shimadzu) with Cu Kα radiation. The thermal behavior was determined by a simultaneous thermal analyzer (Beijing HengJiu Science Instrument Factory, HCT-3) in a corundum

Figure 2. *Desulfurization slag*

Table 1. *Compositions of raw material /wt%*

Raw material	CaO	Fe_2O_3	Al_2O_3	SiO_2	MgO	TiO_2	S	Na_2O	As_2O_3
Desulfurization slag	42.33	28.25	6.86	6.2	4.51	3.05	1.35	0.269	-
Desulfurization residue leached by ammonium chloride	28.26	34.18	9.00	8.33	4.10	3.50	0.68	0.043	-
High alumina coal ash	3.02	1.66	49.90	35.02	0.817	1.71	0.129	0.843	0.0158
Glass cullet	8.95	0.33	0.83	76.01	3.20	-	-	9.85	-

crucible under the protection of atmospheric argon.

$$X_{Fe} = M_{Fe} / M_{TFe} \times 100\% \tag{1}$$

$$M_{TFe} = M_{Fe} + M_{RTFe} \tag{2}$$

3. Design of glass composition

To effectively separate the slag and iron from the desulfurization slag, the melting point of the reduced slag should be low except for the reduced metal iron, and the reduced slag after the recovery of iron should be directly used to prepare basic glass and glass-ceramics. Many studies have shown that Factsage is very effective in studying the reduction process of high-temperature slags [18-20], and the reduced slag is a typical CaO-MgO-SiO_2-MgO (CMAS) system; therefore, Factsage7.2 software was used to calculate the melting point and phases of the system as shown in Fig.3 below, it can be seen from the dotted line in Fig.3 that the melting point of the reduced slag is all below 1300℃, which is conducive to the slag-iron separation. Meanwhile, the phase of the reduced slag in this region is diopside. In view of the mechanical strength, wear resistance and chemical stability of diopside glass-ceramics, it is selected as the target main crystal phase in this paper. In addition, because of the glass cullet containing a certain amount of sodium, a nepheline is easy form during crystallization, which is beneficial to keep the molten state of vitreous body, and the two phases (diopside and nepheline) can better play the advantages of glass-ceramics [21]. Therefore, diopside and nepheline are determined as the main crystal phases of the glass-ceramics in this paper. To make full use of DRLAC and combine with its component characteristics, the DRLAC, glass cullet and high aluminum coal ash were mixed as shown in Table 2. There were four experimental samples in total and the content of reduced carbon was 3% of the total weight.

4. Results and analysis

It can be seen in Fig.4 that the separation of slag and iron after carbon thermal reduction reaction is very good, and a relatively regular cake shaped iron block was obtained. The testing and the calculation of iron also indicate that the recovery rate of iron in desulfurization slag is higher than 99%. XRD spectra of reducing slag in Fig.5 show that the main

Table 2. *The formula for the DRLAC, glass cullet and high aluminum coal ash*

	DRLAC	High aluminum coal ash	Glass cullet
Sample 1	58.5%	4.5%	37%
Sample 2	58.5%	5.5%	36%
Sample 3	58%	5.5%	36.5%
Sample 4	57%	6%	37%

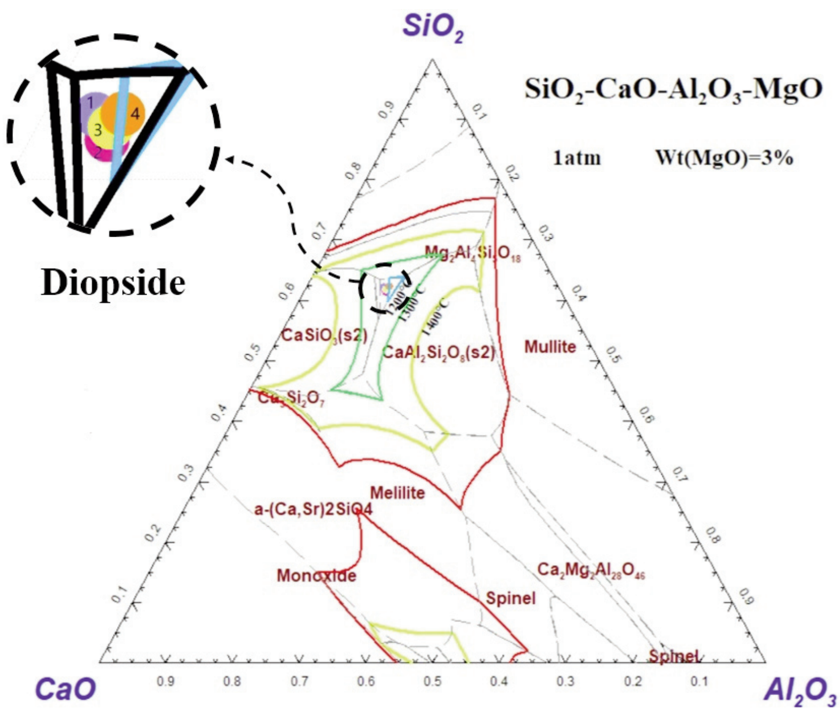

Figure 3. *Melting point and phases of slag in the system of CaO-MgO-SiO_2-MgO*

crystalline phase is diopside and the secondary crystalline phase is nepheline.

The reducing slag was heated to molten state and then homogenized to prepare basic glass, to simulate the high-temperature melting slag directly preparing basic glass and glass-ceramics, the macroscopic photos are shown in Fig. 6.

The glass-ceramic process of basic glass usually includes two processes, namely the formation of crystal nucleus (endothermic process) and the growth of crystal (exothermic process). Therefore, endothermic peaks and exothermic peaks appear in the differential temperatures in DSC curve. As can be seen from the DSC curve in Fig.7, sample 1 has an endothermic peak around 985 K, and two exothermic peaks around 1040 K and 1155 K. In comparison, the endothermic peaks are not obvious in sample 2, 3, and 4; in addition, the temperatures of former exothermic peak drop to about 1030 K and the intensity of the peaks also becomes remarkably smooth, even indistinguishable, while the latter exothermic peak also drops to around 1130 K and the intensity of the peaks was enhanced. The DSC curve indicated that two crystal phases might precipitate in each of the four samples, and the crystal phase near 1130 K is more than that near 1030 K for sample 2, 3, and 4.

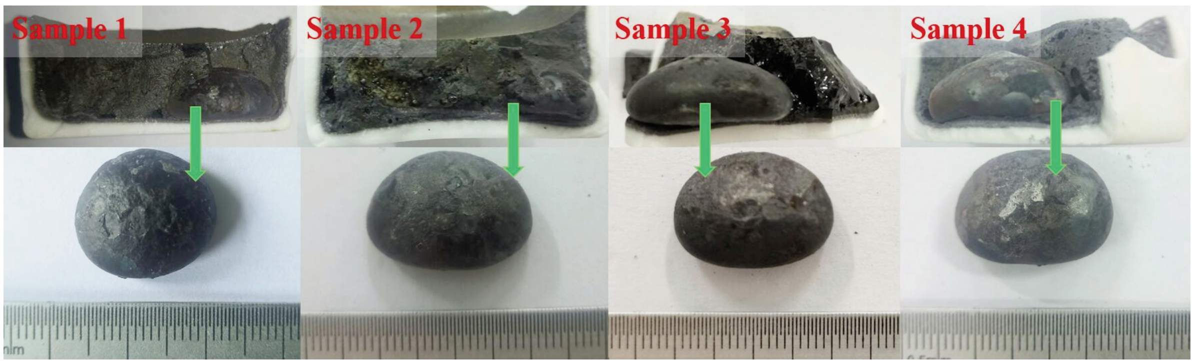

Figure 4. *The separation macrograph of slag and iron after carbon thermal reduction reaction*

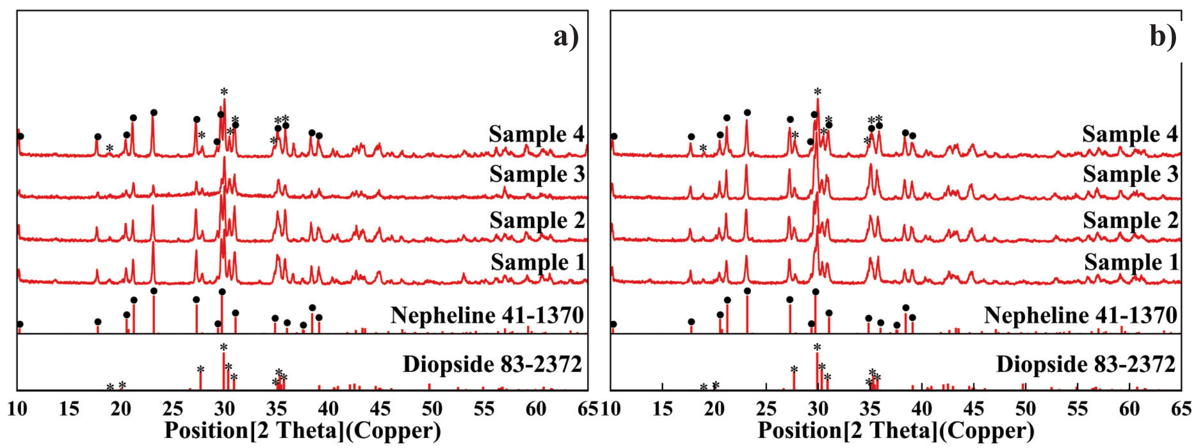

Figure 5. *XRD patterns of reduction slag and glass-ceramics: (a) reduction slag, (b) glass-ceramics*

Figure 6. *The macrograph of basic glass and glass-ceramics*

With the increase of heating rate, both the initial crystallization temperature and the peak temperature of the four samples increased gradually, which was mainly caused by the fact that the heat cannot be supplied in time with the increase of heating rate [22]. From the position of endothermic and exothermal peak, these two temperatures are far lower for that the glass-ceramic's nucleation and crystallization temperatures of metallurgical slag-based are usually about 900 and 1100 ℃ [21], respectively. The formation of nepheline crystals is conducive to reducing the heat treatment temperature and reducing the process energy consumption.

Based on the DSC curves of the four samples, it was determined that the nucleation was carried out at 1033 K for 2 h, and the crystallization was carried out at 1138 K for 2 h, with a heating rate of 5 °C/min, and the glass-ceramics obtained after the heat treatment are shown in Fig. 6. XRD analysis of the glass-ceramics in Fig. 5 shows that the crystalline phases are diopside and nepheline, which is consistent with the XRD spectra of the reduction slag and the results of the two exothermic peaks in the DSC curve. However, from crystal phase intensity of diopside and nepheline, the difference between the four samples was small, which seemed to be not consistent with the characteristics of DSC curves. In fact, 1033 K was not only the formation point of crystal nucleus, but also the growth point of nepheline near 1030 K. In other words, after holding for 2 h at 1033 K in heat treatment, it also promotes the crystallization of nepheline.

The glass-ceramics is obtained by the controlled crystallization of the basic glass after heat treatment, which is a non-uniform nucleation process. At present, classical theories on the crystallization behavior of glass-ceramics mainly include Kissinger equation (Equation 3), Ozawa equation (Equation 4) and Augis-Bennett equation (Equation 5). In this article, these three formulas were used to calculate the crystallization kinetics of glass-ceramics under non-isothermal conditions. The crystallization activation energy E was used to represent the potential barrier needed to overcome the structural rearrangement during the transition from glassy state to crystal state, that is, the difficult degree of crystallization, and the crystal growth index n was used to judge the growth form of crystal.

$$ln\left(T_p^{\,2}/\alpha\right) = E/\left(R\times T_p\right) + ln\left(E/R\right) - lnv \tag{3}$$

$$ln\left(1/\alpha\right) = E/\left(R\times T_p\right) + C \tag{4}$$

$$ln\left(T_p/\alpha\right) = E/\left(R\times T_p\right) + lnK_0 \tag{5}$$

$$n = \left(2.5\times R\times T_p^{\,2}\right)/\left(\Delta T\times E\right) \tag{6}$$

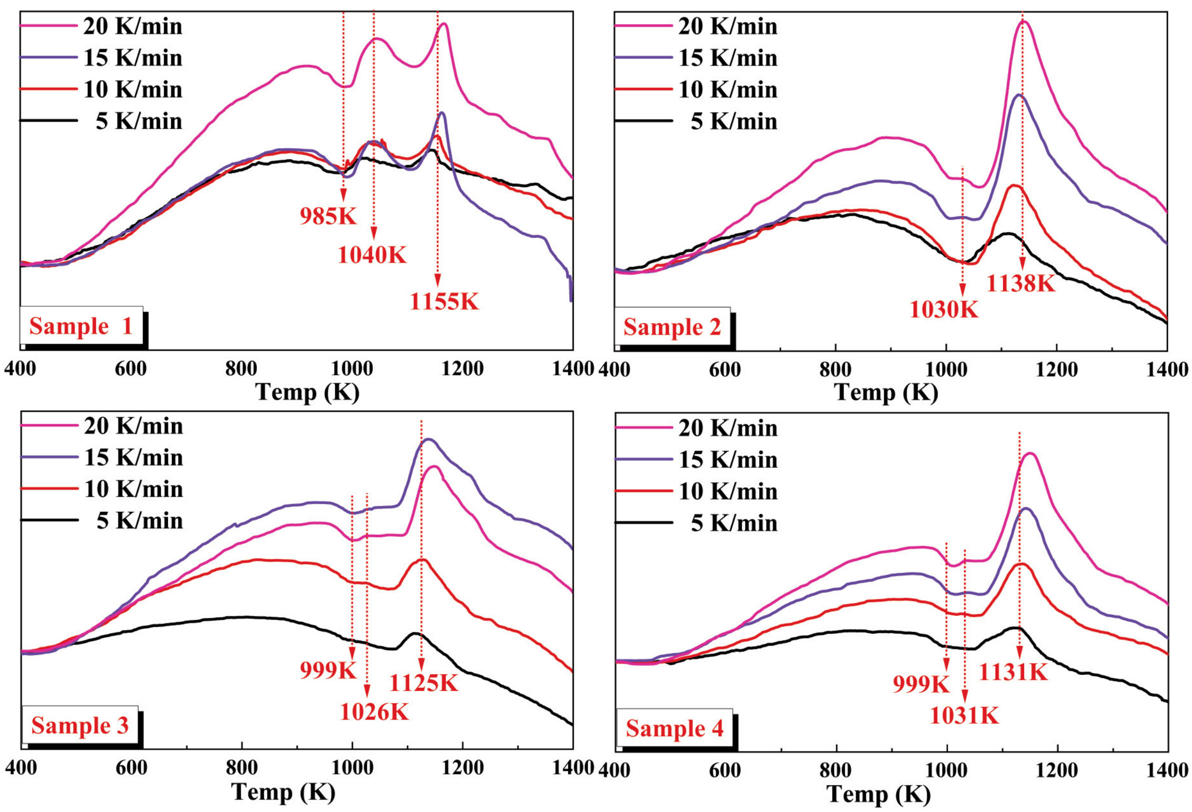

Figure 7. *The DSC curve of different basic glass sample*

where α is heating rate, K/min; Tp is the peak temperature of crystallization,K; v is the frequency factor, C is constant, ΔT is the half peak width of the crystallization peak, K.

According to the DSC curves of the four samples at different heating rates, combined with the Kissinger equation, Ozawa equation and Augis-Bennett equation, the linear fitting was carried out with 1/Tp as the X-axis and $\ln(Tp^2/\alpha)$, $\ln(1/\alpha)$ and $\ln(Tp/\alpha)$ as the Y-axis, respectively. The linear fitting of dynamics was obtained in Fig. 8.

Then Tp, α and R are substituted into the equation to obtain the crystallization activation energy E, as shown in Table 3. By comparing and analyzing the crystallization activation energy obtained by different methods, it can be found that there is little difference between results obtained by the three methods. The value of Ozawa method is relatively high, while the value of Kissinger method is relatively low. Compared with the four samples, the crystallization activation energy of sample No. 3 is the smallest, that is, sample No. 3 has the lowest potential barrier needed to overcome the structural rearrangement and is easier to crystallization. The crystal growth index was calculated by using the Augis-Bennett equation (Equation 6). By substituting the E values of different crystallization activation energies calculated in Table 3 into the equation, and combining with the DSC curve, the crystal growth index n shown in Table 4 can be obtained. It can be seen that the crystal growth index is not very high, which may be due to the fact that there are few oxides with nucleation function, such as TiO_2, in the raw material under the condition of not adding nucleating agent in this paper, and two kinds of crystals are precipitated during heat treatment, which results in mutual interference in the

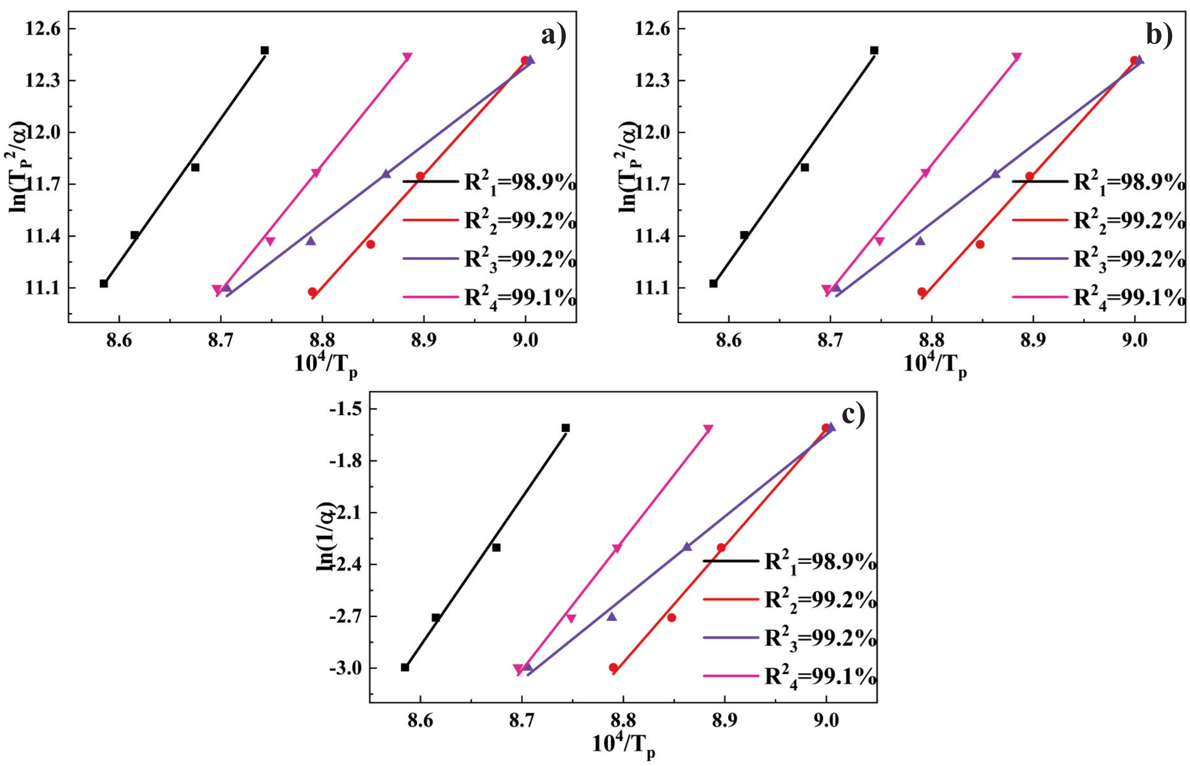

Figure 8. *Linear fitting of dynamics: (a) Kissinger, (b) Ozawa, (c) Augis-Bennett*

Table 3. *Crystallization activation energy of different samples*

Sample	T_p(K)				Crystallization activation energy E（KJ/mol）			
	5	10	15	20	Kissinger	Ozawa	Augis-Bennett	Average
1	1143.75	1152.75	1160.75	1164.85	692.5	711.7	702.2	702.1
2	1111.15	1124.05	1130.25	1137.65	542.3	561.0	551.7	551.7
3	1110.55	1128.35	1137.85	1148.65	374.4	393.2	383.8	383.8
4	1125.65	1137.15	1143.05	1149.95	607.3	626.2	616.8	616.8

Table 4. *Crystal growth index of different samples*

Sample	Heating rate	Half peak width ΔT	Kissinger	Ozawa	Augis-Bennett	Average
1	5	24.1	1.6	1.6	1.6	1.6
	10	24.0	1.7	1.6	1.6	1.6
	15	23.0	1.8	1.7	1.7	1.7
	20	28.1	1.4	1.4	1.4	1.4
2	5	28.1	1.7	1.7	1.7	1.7
	10	47.1	1.0	1.0	1.0	1.0
	15	46.1	1.1	1.0	1.0	1.0
	20	52.8	0.9	0.9	0.9	0.9
3	5	24.7	2.8	2.7	2.7	2.7
	10	34.6	2.0	2.0	2.0	2.0
	15	34.8	2.1	2.0	2.0	2.0
	20	44.4	1.6	1.6	1.6	1.6
4	5	40.6	1.1	1.0	1.1	1.1
	10	41.7	1.1	1.0	1.0	1.0
	15	42.5	1.1	1.0	1.0	1.0
	20	46.9	1.0	0.9	1.0	1.0

initial nucleation stage and weakened crystallization ability of glass. Among them, the n value of sample No. 3 is the largest at the heating rate of 5 K/min. The crystallization mode of the glass-ceramics is changed from two-dimensional to one-dimensional volume crystallization [22], indicating that the preparation of glass-ceramics in this paper needs to add nucleating agent.

5. Conclusion

This paper creatively proposes a new process with desulfurization slag leached by ammonium chloride as pretreatment The results show that iron recovery rate is over 99%, and diopside and nepheline glass-ceramics are produced, which shows that the new process is feasible. However, the crystal growth index is less than 3, which means that the crystallization capacity of the glass-ceramics is low, and nucleating agent is needed in the preparation of glass-ceramics.

1) A new process for preparing commercial calcium carbonate, glass-ceramics and recovering iron from desulfurization slag with ammonium leaching is proposed, and the main point of this paper lies in the processing of desulfurization residue leached by ammonium chloride. Through component analysis, the formula of reduction slag is adjusted to facilitate the separation of iron and slag, and can be used to produce glass-ceramics directly.

2) The carbon thermal reduction experiment can make the iron recovery rate over 99%, and the reducing slag can be transformed into glass-ceramics whose main crystalline phases are diopside and nepheline by homogenization and heat treatment, which show that the process is feasible.

3) The kinetic study of glass-ceramics shows that the crystallization activation energy ranges from 383.8 to 616.8 kJ /mol, which is relatively high, and the crystal growth index is less than 3, therefore, the preparation of glass-ceramics requires the addition of nucleating agent, and the waste residue ricing nucleating agent will be used as the formula material in the next stage of the study.

Author’s contributions

J.-T. Sun: S.-C. Liu, W. Zhang, M.-L. Kuang: experiment, test and validation; Z.-B. Tong: conceptualization, writing–original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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PROUČAVANJE NOVOG PROCESA DOBIJANJA ŽELEZO I NJEGOVE KINETIKE I PRIPREMA STAKLOKERAMIKE IZ ŠLJAKE PROCESA ODSUMPORAVANJA

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Apstrakt

Ovaj rad predlaže novi kreativni proces sa šljakom iz procesa odsumporavanja izluženom amonijum hloridom kao predtretmanom, a glavna cilj ovog rada leži u preradi ostatka odsumporavanja luženog amonijum hloridom. Analizom komponenti formula je prilagođena visokom sadržaju aluminijumskog pepela i staklenog krša, tako da je tačka topljenja redukcione šljake oko 1200 ℃, što olakšava odvajanje železa i šljake. Istovremeno, redukcija šljake se prilagođava ciljnoj fazi kristalizacije, tako da se visoko temperaturno redukovana šljaka nakon termičke redukcije ugljenikom može koristiti za direktnu proizvodnju staklokeramike. Rezultati pokazuju da je stopa dobijanja gvožđa preko 99%, a proizvodi se diopsidna i nefelinska staklokeramika, što pokazuje da je novi proces izvodljiv. Međutim, indeks rasta kristala je manji od 3, što znači da je kapacitet kristalizacije staklokeramike nizak, a agens za nukleaciju je potreban u pripremi staklokeramike.

Ključne reči: *Šljaka procesa odsumporavanja; Staklokeramika; Diopsid i nefelin; Kinetika; Factsage softver*