Materials Research Express

PAPER

Relationship between thermophysical properties and structure of CMAS glass melts based on chromium slag

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Keywords: chromium slag, viscosity, electrical conductivity, surface tension, melt density

Abstract

In an attempt to determine the relationship among the network structure, viscosity, electrical conductivity, surface tension, and density of chromium-containing glass melts, the variation in glass melt thermophysical properties of the CaO–MgO–Al\textsubscript{2}O\textsubscript{3}–SiO\textsubscript{2} quaternary system were investigated by comprehensive physical property analysis and Raman spectroscopy. The results demonstrated that the network structural units \(Q^n\) \((n = 0, 1, 2, 3)\) coexisted in the glass melts and their mutual transformation caused changes in the depolymerization degree, leading to a range of viscous activation energies, conductive activation energies, surface tension, and melt densities. Additionally, the logarithm of viscosity varied linearly with the logarithm of electrical conductivity (slope: 0.67–1.54). Meanwhile, the surface tension and melt density decreased with temperature. Consideration of these interrelations is indispensable during the development of chromium-containing slag glass ceramics, particularly for pilot and industrial applications.

1. Introduction

Chromium slags are hazardous solid wastes generated in the metallurgical industry during chromate production, particularly metallic chromium. Considerable amounts of chromium slags, which are generated by extensive chromium-based industrial processes, are discarded (approximately 4.5 × 10\textsuperscript{7} t). The annual emission has reached 0.8 × 10\textsuperscript{7} t; most of it is disposed via landfill without further management [1, 2]. The large-scale discharge of chromium slags has caused extensive usage of land resources. In addition, the hexavalent chromium (Cr\textsuperscript{6+}) present in waste slags is carcinogenic to humans; it easily leaks into rainwater, thereby, contaminating land and groundwater [3]. Therefore, the detoxification and reuse of chromium residues has become an urgent research topic in recent years. The detoxification of Cr\textsuperscript{6+} by redox reactions has been reported to be a suitable method; however, the reaction process is complicated and the stability of detoxified slags varies with the treatment process. High-temperature melting is another highly valuable method for the utilization of chromium slags, wherein Cr\textsuperscript{6+} is reduced to Cr\textsuperscript{3+} and simultaneously solidified in the glass network. CaO, MgO, Al\textsubscript{2}O\textsubscript{3}, and SiO\textsubscript{2} are the main components of slags, tailings, fly ashes, and other industrial wastes. Some of these wastes contain heavy metal elements, such as Cr, Cu, and Zn. Hence, for effective detoxification and utilization of industrial residues, it is imperative to study the effect of components of aluminosilicate glass on the thermophysical properties, microstructure, and crystallization mechanism of the glass [4–6]. The development of slag-based aluminosilicate glass ceramics (polycrystalline composites prepared from vitreous glass by a series of heat treatments) is considered an efficient method to promote the utilization of residues in comparison with the conventional methods. Aluminosilicate glass ceramics exhibit excellent characteristics, such as high
mechanical strengths, adjustable thermal expansion coefficients, superior corrosion resistance, and high thermal shock resistance. These remarkable physicochemical properties make Al$_2$O$_3$–SiO$_2$ glass ceramics highly suitable for various applications: abrasion and thermal insulation layer materials, sealants, and building and decoration materials. Therefore, the development of slag-based aluminosilicate glass ceramics can promote the utilization of slags; moreover, the application of these glass ceramics can enhance the product value and result in considerable economic benefits. Moreover, the composition of chromium slags is similar to that of glass ceramics. For instance, Bai et al studied the devitrification of Cr-containing slag glass ceramics, demonstrating it to be an appropriate route to synthesize glass ceramics [7]. Zhao et al studied the effect of Fe$_2$O$_3$ on the precipitation of spinel and pyroxene in chromium slag-based glass ceramics and found that a high amount of iron oxide promoted the enrichment of chromium in the spinel phases [8]. Liao et al synthesized glass ceramic products and achieved a maximum chromium partitioning ratio of 90% in the spinel phase, demonstrating that spinel glass ceramics successfully immobilize chromium [9]. In addition, we found that the melt thermophysical properties significantly affected the melting process, particularly the manufacturing process, and performance of glass ceramic samples.

The melt thermophysical properties, including viscosity, surface tension, electrical conductivity, and density, are essential and fundamental properties that considerably affect the formation and performance of molten glass, particularly in pilot and industrial applications. Cochain et al investigated the effects of structure and composition on the atomic mobility by measuring the viscosity and electrical conductivity, which affected the kinetics of the redox reactions [10]. This fundamental study provided an appropriate method for the molding of glass melts. Ehrt and Keding showed that the electrical conductivity of sodium borosilicate glass melts increases in the softening region, assuming that [BO$_4$]$^-$ units transformed into [BO$_3$O$_2$]$^-$ units, leading to a drastic increase in the electrical conductivity [11]. Moreover, several studies have found that the dripping behavior is affected by the density and surface tension of molten glass, and that the packing ratio related to the density reflects the structural information of glass melts [12–14]. Generally, electrical conductivity is restricted by ion concentration and migration resistance, and the latter is mainly affected by the degree of polymerization, indicating a subtle relation between viscosity and electrical conductivity [15]. Although the viscosity and electrical conductivity of the CaO–MgO–Al$_2$O$_3$–SiO$_2$ (CMAS) quaternary system have been extensively studied, the quantification relation between these two properties has not been properly investigated [16–19]. Notably, the main constraining factor is the content of Al$_2$O$_3$ in the CMAS system, which affects not only the cationic concentration, but also the polymerization degree, thereby causing a complex influence on the conductivity [20]. Most studies on the melt thermophysical properties are mainly focused on slags and metal separation in the metallurgical industry, and the comprehensive utilization of classified slags, particularly for the development of materials such as glass ceramics, has received considerably less attention.

In this study, molten glass was synthesized using chromium slag, and the effect of the melt basicity, which was modified by varying the amount of slag used for preparation of base glass, on the thermophysical properties of the glass was studied. In addition, the melt structure was investigated by Raman spectroscopy, on the basis of the Q$^n$ unit concept, where $n$ (= 0, 1, 2, 3, 4) is the number of non-bridging oxygen (NBO) atoms coordinated to Si, which relates the melt viscosity and degree of polymerization of the glass network [21]. Furthermore, the relationship between the thermophysical properties and glass network structural units was investigated, aiming to provide a theoretical basis for the formation of chromium-containing slag glass ceramics.

### 2. Experimental

The basic material used in this study was chromium slag, and its main components are listed in table 1. The x-ray diffraction pattern of the slag is shown in figure 1, indicating that the slag mainly comprised iron silicate Fe$_2$SiO$_4$ (JCPDS 80–1625) and glass phases.

| Cr-slag | SiO$_2$ | CaO | MgO | Al$_2$O$_3$ | TFe | K$_2$O | Na$_2$O | TiO$_2$ | BaO | MnO | P | F | Cr | Total |
|--------|--------|-----|-----|-------------|-----|-------|--------|--------|-----|-----|---|---|----|-------|
|        | 26.11  | 29.17| 3.89| 13.99       | 14.42| 0.39  | 5.21   | 0.24   | 0.50| 0.34| 1.46| 3.86 | 0.50 | 100   |

The contents of the base glass are listed in table 2. For the synthesis, typically, the raw materials were ground and sieved through a 200-mesh, weighed, and mixed for 1 h to achieve homogeneity. Then the mixtures were heated to 1723 K for 3 h in a high-temperature furnace. Subsequently, the glass melts were quenched, dried, and sieved to generate samples for melt thermophysical property measurements. All tests were performed using a melt physical tester (RTW-1002, China), a diagram of which is shown in figure 2.

The tester comprised three modules: a torque sensor, a conductometer, and an electric balance for viscosity, melt electrical conductivity, and melt surface tension and density measurements, respectively. Typically, 150 g of...
the sieved sample was placed in a molybdenum crucible and reheated. The heating system is shown in figure 3. The thermophysical properties were measured at various temperatures: 1653, 1683, 1713, 1743, and 1773 K. In the second stage (cooling stage), the viscosity of the glass melts was determined. Then, the thermophysical properties in the heating stage and viscosity-temperature relationship in the cooling stage were simultaneously determined. The melt viscosity was measured by the rotational method with a molybdenum rotor, which was connected to a torque sensor with an alumina rod. For the measurements, the rotor was immersed 30 mm into the melt. The electrical conductivity was measured with a pair of molybdenum electrodes connected to a conductometer. During the measurements, N2 was flushed into the furnace to inhibit oxidation of the molybdenum crucible and test elements at high temperatures. In addition, water was circulated in the interlayer to cool the furnace body.

3. Results and discussion

3.1. Relationship between melt viscosity and structure

The viscosities of the glass melts in the cooling stage are shown in figure 4. Pioneering studies have demonstrated that viscosity varies with temperature and composition. The viscosity-temperature relationship can be described using the Arrhenius equation, as follows [22–25]:

\[
\eta = A \exp\left(\frac{E_a}{RT}\right)
\]

where \( \eta \), \( E_a \), \( A \), \( R \), and \( T \) are the viscosity, viscous activation energy, Arrhenius constant, absolute gas constant (8.314 J (mol·K)), and thermodynamic temperature, respectively.

Equation (2), obtained by taking the logarithm of equation (1),

\[
\ln \eta = \ln A + \frac{E_a}{RT}
\]

indicates that the relationship between \( \ln \eta \) and \( 1/T \) is linear. The fitting curves and related parameters are presented in figure 5 and table 3, respectively.

As depicted in figure 4, the melt viscosity increased as the temperature decreased from 1770 K to 1690 K. Additionally, with an increment in the melt basicity from 0.19 to 0.25, the melt viscosity initially increased, and

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Table 2. The main compositions of base glasses.

| Sample No. | CaO  | MgO  | Al₂O₃ | SiO₂  | Na₂O  | Basicity |
|------------|------|------|-------|-------|-------|----------|
| ZJ1        | 18.28| 2.61 | 9.41  | 65.55 | 4.15  | 0.19     |
| ZJ2        | 18.24| 2.99 | 10.16 | 63.99 | 4.63  | 0.22     |
| ZJ3        | 18.44| 3.12 | 10.41 | 62.90 | 5.13  | 0.23     |
| ZJ4        | 19.98| 3.26 | 10.29 | 60.94 | 5.53  | 0.24     |
| ZJ5        | 21.77| 3.33 | 11.20 | 57.92 | 5.78  | 0.25     |

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Figure 1. The XRD analysis of chromium-containing slag.
then decreased at constant temperature. The maximum value of the viscous energy was determined to be 210.29 kJ mol$^{-1}$, as presented in table 3.

The structural units, constituting the melt, migrated from one position to another at the atomic level, leading to melt fluidity. The energy barrier that must be overcome during the migration process is defined as the viscous activation energy, which reflects the ease of flow of the melt structural units. In silicate melts, the AlO$_4^{−}$ and
SiO$_x$ clusters are significantly larger than the other cations in the melt. Therefore, their type and quantity are the main factors that affect the viscosity of the silicate melt.

Raman spectroscopy has been widely reported as an effective tool to analyze melt structures [26–28]. Therefore, the quenched samples were subjected to Raman spectroscopy analysis (figure 6) to explore the changes in the silicate network structure. The Raman spectra mainly comprise three bands in the ranges of 400–600, 650–800, and 850–1200 cm$^{-1}$, which were assigned to the Si-O$_b$-Si bending vibration, Al-O$_{ab}$ symmetrical stretching vibration, and Si-O$_{ab}$ symmetrical stretching vibration, respectively. As depicted in
Figure 6, the weak vibration band at 650–800 cm\(^{-1}\) indicates that the content of Al\(^{3+}\) ions participating in the formation of the glass network structure in the melt was much lower than that of the Si\(^{4+}\) ions. Lee reported that the correlation between bridge oxygen (BO; O\(^0\)), free oxygen (O\(^2-\)), and NBO (O\(^-\)) can be expressed by the following equilibrium [29]:

\[
2Q^- = Q^0 + Q^2^- \quad (3)
\]

and that in silicate melts, the dominant anionic units are formed by Si-O\(_{ab}\) bonds, which are in turn formed by Si\(^{4+}\) via coordination with the NBO (O\(^-\)), because of the higher ion field strength of Si\(^{4+}\) than that of Al\(^{3+}\).

The relation between the Si-O\(_{ab}\) band (850–1200 cm\(^{-1}\)) intensity and the polymerization degree can be illustrated using the Q\(^n\) concept; where \(n = 0, 1, 2, 3, \) and \(4\), corresponding to [SiO\(_4\)]\(^{4-}\), [Si\(_2\)O\(_7\)]\(^{6-}\), [SiO\(_3\)]\(^{2-}\), [Si\(_2\)O\(_5\)]\(^{2-}\), and [SiO\(_2\)] in the silicate melt, respectively. Accordingly, a Gaussian fit was performed on the Raman spectra in the range of 850–1200 cm\(^{-1}\), as shown in figure 7. It could be concluded that only [SiO\(_4\)]\(^{4-}\), [Si\(_2\)O\(_7\)]\(^{6-}\), [SiO\(_3\)]\(^{2-}\), and [Si\(_2\)O\(_5\)]\(^{2-}\) were present in the melts. The deconvolution parameters are presented in table 4. The deconvoluted bands at \(\sim 880, \sim 940, \sim 1020, \) and \(\sim 1145 \text{ cm}^{-1}\) were assigned to [SiO\(_4\)]\(^{4-}\), [Si\(_2\)O\(_7\)]\(^{6-}\), [SiO\(_3\)]\(^{2-}\), and
Table 4. Deconvolution parameters of Raman spectra.

| Sample No. | Q^n units | Center (cm⁻¹) | Amplitude (a.u.) | % Area  | (Q^n)²/Q² | R²   |
|------------|-----------|--------------|-----------------|---------|-----------|------|
| ZJ1        | Q₀        | 880.12       | 1359.87         | 3.37    | 0.05      | 0.9994 |
|            | Q¹        | 941.51       | 6383.10         | 29.35   |           |       |
|            | Q²        | 1023.28      | 8790.48         | 65.48   |           |       |
|            | Q³        | 1160.96      | 484.10          | 1.78    |           |       |
| ZJ2        | Q₀        | 884.97       | 1795.84         | 5.45    | 0.16      | 0.9993 |
|            | Q¹        | 944.04       | 6161.85         | 30.18   |           |       |
|            | Q²        | 1023.10      | 7650.98         | 61.26   |           |       |
|            | Q³        | 1148.11      | 617.37          | 3.09    |           |       |
| ZJ3        | Q₀        | 884.37       | 1344.46         | 6.10    | 0.36      | 0.9991 |
|            | Q¹        | 943.58       | 4355.28         | 30.98   |           |       |
|            | Q²        | 1019.27      | 5131.92         | 58.36   |           |       |
|            | Q³        | 1130.87      | 590.86          | 4.55    |           |       |
| ZJ4        | Q₀        | 884.10       | 2102.02         | 8.21    | 0.17      | 0.9991 |
|            | Q¹        | 943.96       | 5580.43         | 32.38   |           |       |
|            | Q²        | 1018.86      | 6270.01         | 56.29   |           |       |
|            | Q³        | 1129.67      | 514.57          | 3.09    |           |       |
| ZJ5        | Q₀        | 884.62       | 786.69          | 3.75    | 0.06      | 0.9986 |
|            | Q¹        | 942.40       | 4299.60         | 42.87   |           |       |
|            | Q²        | 1019.23      | 4120.11         | 51.65   |           |       |
|            | Q³        | 1130.66      | 200.98          | 1.71    |           |       |

\[ [\text{Si}_2\text{O}_5]^{2-} \], respectively. Meanwhile, the Raman band corresponding to Q¹ could not be deconvoluted because its content was low.

It could be concluded from the fitting results that the abundance of Q⁰ and Q³ initially increased, and then, decreased as the basicity increased; meanwhile, the relative content of Q¹ units exhibited an upward trend. The units predominantly formed, corresponding to the degree of polymerization of the glass melts (NBO/T < 1.0) can be described by the following equilibrium [30]:

\[
2Q³ \rightleftharpoons Q² + Q⁴ \Rightarrow [\text{Si}_2\text{O}_3]^{2-} \rightleftharpoons [\text{SiO}_2]^2- + [\text{SiO}_2]
\]  

(4)

The equilibrium constant \( K \) for equation (4) can be defined as follows:

\[
K = Q² \cdot Q⁴ / (Q³)^2
\]  

(5)

\[
Q⁴ = K \cdot (Q³)^2 / Q² \propto \text{polymerization degree}
\]  

(6)

The constant \( K \) determines the degree of polymerization; the lower the value of \( \ln K \), the higher is the degree of depolymerization. The \( (Q³)^2 / Q² \) ratios are listed in table 4; the ratio initially increased and then decreased, indicating that the polymerization degree of the aluminosilicate glass melt first increased and then decreased as the melt basicity increased. Therefore, a higher viscous activation energy was required for the migration of the structural units, which reached 210.29 kJ mol⁻¹ for sample ZJ3.

3.2. Effect of slag content on glass melt electrical conductivity

The electrical conductivity of glass melt is a parameter that reflects the ease of charge flow in the melt under high-temperature conditions, and is one of the basic physical properties of the melt. Therefore, the determination of melt electrical conductivity is highly significant in the production of electrically insulating materials using glass ceramics. In this study, the electrical conductivity was measured in the heat-retaining interval (figure 3), during which the viscosity was measured at 1653, 1683, 1713, 1743, and 1773 K. The electrical conductivity values for the glass melts are presented in figure 8; as observed, ZJ3 exhibited the lowest value.

In melts, the cations conduct electricity, and the cation mobility increases with temperature [31]. Accordingly, the melt electrical conductivity continuously increases with temperature. Furthermore, most transport properties can be predicted using the Arrhenius equation, which links the structure, properties, and temperature.

The relationship between the electrical conductivity and temperature can be described by the Arrhenius equation, as follows [32]:

\[
\kappa = B \exp(E_a / RT)
\]  

(7)

where \( \kappa, B, E_a, R \) and \( T \) correspond to the electrical conductivity, Arrhenius constant, electrical activation energy, absolute gas constant (8.314 J (mol · K)), and temperature, respectively.
The fitting data for the electrical conductivity and viscosity ($T = 1653, 1683, 1713, 1743, \text{and} 1773 \text{ K}$) are shown in figures 9 and 10, respectively. The conductance activation energy firstly decreased, and then, increased. Meanwhile, compared to ln $\kappa$, ln $\eta$ exhibited an opposite trend as a function of temperature. The electrical conductivity depended on the cation mobility and amount of SiO$_{n}$ in the silicate melt, i.e., the polymerization degree, as previously discussed. According to the Raman results, structural units with various ionic radii and bond strengths were formed in the networks: monomers, dimers, chains, and sheets \cite{33, 34}. As observed from figures 9 and 10, the variation trend of the conductance activation energy agreed with that of the degree of depolymerization, as the complicated network structural units with high polymerization degree suppressed the mobility of cations, particularly Ca$^{2+}$.

On the basis of equations (1) and (4), the relation between viscosity and electrical conductivity can be described as follows \cite{35}:

$$\ln \eta = c - n \ln \kappa$$  \hspace{1cm} (8)

where $n$ is the ratio of the viscous activation energy to the conductance activation energy, and $c$ is a constant. The relation between ln $\eta$ and ln $\kappa$ for the CMAS system is shown in figure 11. As revealed by figure 11, this system could be well described using equation (8). However, $n$ varied with the composition of the base glass, particularly with the valences and types of metal cations. With increasing amount of slag, the changes occurring in the network structure in the glass melt led to changes in the viscosity, electrical conductivity, and activation energy.
of the melt, which changed the ion transport mechanism. At high viscous activation energies, the alkali metal ions in the melt conducted electricity via the ion transport mechanism, while at low viscous activation energies, they conducted electricity via the diffusion mechanism, thereby causing fluctuations in $n$.

3.3. Effect of slag content on glass melt surface tension and density

The melt surface tension ($\gamma$) and melt density ($\rho$) are considered important properties in glass manufacturing because of their effects on melting, clarification, and homogenization, particularly on the adhesion between glass melts and metal surfaces, which is critical in the preparation of glass ceramic-metal composite materials.

The surface tension values for the chromium slag glass melts are depicted in figure 12(a). As observed, the surface tension gradually decreased with increasing temperature, in agreement with the previous report. Notably, a linear relationship was fitted (figure 12(b)); the temperature coefficient ($-d\gamma/dT$) was extracted from the slope of the curve. Moreover, it has been reported that most melt properties are mainly affected by the depolymerization degree, while the cation concentration causes only secondary effects. ZJ3 exhibited the highest polymerization degree (figure 10), and the maximum viscous activation energy. The glass melt with the highest polymerization degree, comprised considerably more BOs comparison with the other samples. Notably, the interaction among the melt surface layers enhanced the surface tension.
The density versus temperature plots for the chromium slag glass melts are shown in figure 13. As observed, for samples, the density linearly decreased with increasing temperature because of thermal expansion. The Raman results confirmed that melt density was mainly determined by the molar volume. AlO\textsubscript{x}\textsuperscript{m−} was gradually formed with the introduction of chromium slag, and the amount of calcium or alkali metal ions was sufficient to induce a charge balance, causing changes in the molar volume of the CMAS melt with the formation of Si–O–Ca bonds [40, 42]. In addition, the molar volume of AlO\textsubscript{x} was compensated by two alkali metal cations (e.g., Na\textsuperscript{+}), was approximately three times that compensated by a divalent cation. Therefore, the molar volume of the system increased as the content of the alkali metal ions and aluminum ions in the melt increased, resulting in a decrease in the melt density with increasing melt basicity.

4. Conclusions

In this study, the variation in the thermophysical properties of CMAS glass melts was explored. The viscosities of the glass melts increased as the temperature decreased. Meanwhile, with increasing melt basicity, the melt viscosity initially increased and then decreased because of the transformation of the structural units. Notably, the maximum viscous activation energy of the glass melt was determined to be 210.29 kJ mol\textsuperscript{−1}. Additionally, the depolymerization degree was investigated by Raman spectroscopy. The peak fitting results indicated that [SiO\textsubscript{4}]\textsuperscript{4−}, [Si\textsubscript{2}O\textsubscript{7}]\textsuperscript{6−}, [SiO\textsubscript{3}]\textsuperscript{2−}, and [Si\textsubscript{2}O\textsubscript{5}]\textsuperscript{2−} coexisted in the glass melts. Unequal concentration of the structural units resulted in various degrees of depolymerization, and consequently, different viscous activation
energies. Furthermore, the relationship between viscosity and melt electrical conductivity was determined in the heating-up stage, and the fitting results illustrated a linear relationship, with n in the range of 0.67–1.54. Finally, the surface tension and density values of the glass melts decreased with increasing temperature, because of the changes in the degrees of depolymerization, and the latter was also affected by the compensating cations present around the AIO₅ units, which enhanced the molar volume of the system, and consequently, reduced the melt density.

Acknowledgments

This research was financially supported by the Natural Science Foundation of Inner Mongolia Autonomous Region (Nos. 2018LH05021 and 2019MS05050), Inner Mongolia University of Science and Technology Innovation Fund (Nos. 2016QDLB29 and 2016QDLB27), Inner Mongolia Autonomous Region university scientific research project (No. NZZJZ18141), and National Natural Science Foundation of China (Nos. 51704172, 51774189, and 11564031).

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