Effect of Al\textsubscript{2}O\textsubscript{3} addition on the non-isothermal crystallization kinetics and long-term stability of BCABS sealing glass for IT-SOFCs

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Effect of Al$_2$O$_3$ addition on the non-isothermal crystallization kinetics and long-term stability of BCABS sealing glass for IT-SOFCs

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Effect of Al$_2$O$_3$ addition on the non-isothermal crystallization kinetics and long-term stability of BCABS sealing glass for IT-SOFCs

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Abstract: Owing to adjustable thermal expansion performance, BaO–CaO–Al$_2$O$_3$–B$_2$O$_3$–SiO$_2$ (BCABS) glass has a promising commercialization prospect for intermediate temperature-solid oxide fuel cells (IT-SOFCs) sealing. Herein, Al$_2$O$_3$ with two different contents was added into the same glass formulation, referred to as A and B glass, respectively. In terms of the non-isothermal crystallization kinetic behavior, the effect of Al$_2$O$_3$ as the unique intermediate was innovatively studied on the long-term performance of BCABS sealing glass. After the heat treatment at 1023 K for 100 h, the change of the network structure and the expansion coefficient of the glass were characterized. The results showed that the addition of Al$_2$O$_3$ as a network forming body could enhance the structure of glass, and increase the activation energy for glass transition, which could effectively inhibit the crystallization ability of sealing glass. Therefore, the B glass with the higher Al$_2$O$_3$ content showed the better long-term sealing ability, which was greatly beneficial for IT-SOFCs sealing.

Keywords: intermediate temperature-solid oxide fuel cell (IT-SOFC); BaO–CaO–Al$_2$O$_3$–B$_2$O$_3$–SiO$_2$ (BCABS) sealing glass; Al$_2$O$_3$; non-isothermal crystallization kinetics; long-term stability

1 Introduction

Intermediate temperature-solid oxide fuel cells (IT-SOFCs) as electrochemical devices have great potentials in the stationary and mobile applications due to the high-efficiency system of power generation [1,2]. IT-SOFCs could obtain the higher power density by electrochemical reactions, which directly convert hydrogen or hydrocarbon into electricity without any moving parts at the temperature range of 600–800 °C [3]. According to the stacking design and fabrication, the planar type and sealless tubular design for IT-SOFCs have been given much favor by researchers at present [4]. The sealless tubular design has a relatively long current path through the cell, which would result in the great resistive loss. As compared to the tubular design, the planar-type IT-SOFCs could provide the higher power density due to the effective planar structure that consists of the repeated unit by the combination of anode–electrolyte–cathode structure and interconnector [5]. The suitable sealing materials must be required to prevent fuel leakage and air mixing at the high operating temperature to create a hermetic, rugged, stable, and safe stack, which is the urgent

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requirement of the planar-type IT-SOFCs [6].

The sealing materials must simultaneously meet several requirements as follows: gas tightness, electrically insulation, suitable viscosity and chemical inertness, thermal expansion coefficient (TEC) compatibility, and good adherence with other cell components [7]. Sealing materials can be classified in two categories: (1) compressive seals such as metallic or mica-based, (2) rigid seals such as glass, glass-ceramics [8]. Because glass and glass-ceramic sealants exhibit strong bonding to the interface, many studies have been dedicated to optimize the formulation of glass-ceramics to meet most of the requirements. Through adjusting the formulation of glass, the TEC of glass could be directly changed to effectively avoid the stress caused by the mismatch of TEC among the sealants and other cell components at high temperature. Due to high expansion value and adjustable thermal expansion performance, BaO–CaO–Al2O3–B2O3–SiO2 (BCABS) sealing glass has taken much attention of the researchers [9–12]. However, during the heat treatment condition, the hexagonal celsian phase (TEC = 8.0×10^{-6} K^{-1}) in BCABS glass would be transformed into the monoclinic celsian phase (TEC = 2.3×10^{-6} K^{-1}), which would decrease the TEC of BCABS glass to result in serious cracks along the interface of bonded materials [13]. Ghosh et al. [14] reported that the addition of LaO in BCABS sealants could increase the viscosity of the glass, and then inhibit the glass crystallization at relatively high temperature. Lin et al. [15] indicated that through adjusting the formulation of glass, the self-healing of BaO–Al2O3–SiO2 based glass could fill the cracks caused by the stress under the heat treatment condition.

As seen from the above studies, the two critical parameters for the long-term sealing ability of sealing glass are the thermal expansion coefficient and glass crystallization temperature. In order to obtain a suitable glass sealant, it is essential to elucidate the crystallization kinetics of glass during the heat treatment condition, which is a key subject for researching glass crystallization and then providing new opportunity for structure control by innovative design and processing techniques. In addition, Al2O3 is the most frequently used intermediate in sealing glass, but almost the only intermediate in BaO–CaO–Al2O3–B2O3–SiO2 (BCABS) glass, which could regulate the thermal properties of the glass well. If Al^{3+} was 4-coordinated, it would participate into the glass network and enhance the structure symmetry of glass [16]. Dai et al. [17] observed that increasing the aluminum content from 0 to 30 wt% in the Al2O3-based compressive seals decreases the leakage rate and increases the thermal cycle stability. Wang et al. [18] investigated the viability of the seal and found that the sealing performance of the seal is significantly improved by increasing the Al2O3 content and the seal containing 50 wt% Al2O3 shows the best sealing performance among the studied compositions. However, the role and the related influence of Al2O3 on the structure and crystallization dynamics of sealing glass have been poorly investigated. The purpose of this article was to study the influence of Al2O3 addition on the thermal performance of BCABS glass from the aspect of the non-isothermal crystallization kinetics.

## 2 Experimental

The BCABS sealing glass was prepared based on preliminary preparation [19], and the raw compositions of A and B glass sealants with different Al2O3 additions are listed in Table 1.

The raw materials composed of SiO2 (analysis reagent, AR), Al2O3 (AR), H3BO3 (AR), BaCO3 (AR), Na2CO3 (AR), and CaCO3 (AR), were wet-mixed and melted in the high temperature chamber furnace in air. The molten glass was quenched, and then re-milled in alcohol (AR) for 4 h in a planetary ball miller (QM-3SP2). One part of pulverized glass was subsequently subjected to prepare the glass slab for the determination of the TEC, the glass transition temperature (Tg), and softening temperature (Ts) by dilatometric measurements (Dilatometer 402E NETZSCH, heat rating: 10 K/min). Through pressing the powders containing the polyvinyl butyral (PVB) binders into strips by the laminating machine (769YP-15A), the strips were obtained with

### Table 1 Component ration of A and B glass

| Component | Mole ration of A glass (mol%) | Mole ration of B glass (mol%) |
|-----------|-------------------------------|-------------------------------|
| SiO2      | 28.52                         | 28.52                         |
| Al2O3     | 4.20                          | 10.31                         |
| H3BO3     | 25.23                         | 25.23                         |
| BaCO3     | 28.46                         | 28.46                         |
| CaCO3     | 12.48                         | 12.48                         |
| Na2CO3    | 1.10                          | 1.10                          |
| Total     | 100                           | 106.11                        |

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the dimensions of 5 mm × 5 mm × 20 mm. The dry-presssed rectangular glass strips were heat-treated at 1123 K for 30 min in a fast heating furnace, and then conducted through the operating condition at 1023 K for 0 and 100 h. After that, these samples were used for the thermal expansion testing. Other glass powders were heated in differential thermal reanalysis (DTA, STA449C comprehensive thermal analyzer in Netzsch, Germany) at four different heating rates. The heat of transformation and other essential physical quantities were obtained from the thermograms with the help of software. Furthermore, the X-ray diffraction (XRD, Bruker AXS8-Advance) with Cu Kα radiation was used to identify the formed crystalline phases of the quenched glass after different heating treatments. These pellets were recorded by Fourier transform infrared (FTIR) spectra in the range of 1500 to 400 cm⁻¹ using a Perkin Elmer-Spectrum BX with a spectral resolution of 1 cm⁻¹. Each test sample was mixed with KBr in an agate mortar and then pressed into 13 mm diameter pellets.

The half-cell consisted of 8 mol% yttrium-stabilized zirconia (8YSZ) as the electrolyte and NiO/8YSZ as the anode. The above quenched BCABS glass powders with PVB binder were used to prepare plates, and then be pressed on the half-cell to obtain the glass/electrolyte–anode/glass samples with a sandwich structure. The sandwiched samples were firstly heated to the sealing temperature (1123 K) and maintained for 30 min, and then soaked at the operating temperature (1023 K) for 100 h. The microstructure of the interface between the glass layer and anode/electrolyte layer was obtained by using scanning electron microscopy (SEM, JSM-6700 F) techniques, which could be used to evaluate the adhesion of the glass on the electrolyte and anode in the half-cell.

3 Results and discussion

3.1 FTIR investigation

FTIR transmission spectra of A and B glass with different Al₂O₃ addition at room temperature are shown in Fig. 1. The bands of 1393 and 1203 cm⁻¹ are corresponding to B–O–B anti-symmetry stretching vibrations in [BO₃] triangle and [BO₄] tetrahedron, respectively [20]. The anti-symmetry layer stretching vibrations of the bridging oxygen bond Si–O–Si in [SiO₄] tetrahedron and the non-bridging oxygen bond Si–O⁻ in silicate glass cause the absorption bands of 1040 and 950 cm⁻¹, respectively [21]. As shown in Fig. 1, the two absorption bands are combined together into one band of around 973 cm⁻¹ in the FTIR spectra of A and B glass. The absorption band of 468 cm⁻¹ is attributed to bending vibration of Si–O–Si/B–O–B linkage in A glass, which reveals on the band of 468 cm⁻¹ in B glass. The absorption bands of 721 cm⁻¹ in A glass and 726 cm⁻¹ in B glass are corresponding to the stretching vibration of Si–O–Al [22], which verifies that the Al³⁺ ions exist in four-fold coordination in both A and B glass.

3.2 Crystallization kinetic behavior by DTA

DTA has become a convenient and widely used tool for studying the kinetics of phase transformation, and the kinetic data on the phase transformation can be obtained from this technique in either non-isothermal or linear heating (scanning) mode [23]. Long-term thermal and chemical stability of sealing glass depends on the Tg and crystallization temperature (Tc) of glass during sealing [23]. To maintain the hermeticity of glass, Tg value should be below the operating temperature (1023 K) of IT-SOFC to relieve thermal stress and proceed self-healing of cracks, and Tc should be higher than 1023 K to avoid excessive glass flow. Different glass formulation can cause changes in Tg and Tc, which have great influence on the crystallization of glass. The crystallization of sealing glass is a great influence on adhesion between glass and other cell components during joining or operating period [24].

As shown in Fig. 2, the DTA thermograms of A and B glass powders are recorded at the heating rates of 5, 10, 15, and 35 K/min or 10, 15, 25, and 35 K/min,
respectively. There is a small endothermic peak attributed to the glass transition temperature range, and its lowest point refers to \( T_g \). There is subsequently also an exothermic peak originating from the amorphous-crystalline transformation. The characteristic point of exothermic peak is corresponding to the maximum crystallization rate. The values of \( T_g \) and \( T_c \) for A and B glass are listed at different heating rates in Table 2.

The crystallization kinetics of the glass powders was studied using the formal theory of transformation kinetics as developed by Johnson and Mehl [25] and Faleiros et al. [26] as shown below:

\[
\ln \left( \frac{T_p^2}{\alpha} \right) = \frac{E_c}{RT_p} + k_1
\]

where \( \alpha \) is the heating rate, \( k_1 \) is a constant, \( R \) is the gas constant. The relationship between \( \ln \left( \frac{T_p^2}{\alpha} \right) \) versus \( 1000 / T_p \) from the experimental data is plotted as shown in Fig. 3. The slope of this graph gives the activation energy of crystallization (\( E_c \)). The \( E_c \) of A and B glass could be calculated to be 233.89 and 406.03 kJ/mol, respectively. The result indicates that the B glass with the higher \( E_c \) is more difficult to crystallize, which could obtain more stable performance and wider practical application.

The Kissinger method is also valid for the glass transition and has often been used to calculate the activation energy for glass transition (\( E_t \)) by the following relation [27]:

\[
\ln \left( \frac{T_g^2}{\alpha} \right) = \frac{E_t}{RT_g} + k_2
\]

Kissinger’s formula [27] can also be used for the evaluation of the activation energy of the glass transition using the following formula:

\[
\ln \left( \frac{1}{\alpha} \right) = \frac{E_t}{RT_g} + k_3
\]

The values of \( E_t \) calculated from the slopes of the plots (in Fig. 4 and Fig. 5) can be obtained according to formulas (2) and (3). The values \( E_t \) of A and B glass are 396.99 and 594.03 kJ/mol in Fig. 4, respectively. The slope in Fig. 5 shows the values of \( E_t \) of A and B glass are 411.93 and 609.22 kJ/mol, respectively. The activation energies of glass obtained in different methods have been summarized in Table 3. As shown in Table 3, the values calculated by the two methods are approximately equal, which verifies the effectiveness of the two methods. Ray [28] has reported that \( T_g \) is related to the density of covalent cross linking, number and strength of co-ordinate links formed between oxygen atoms and the cations, and the oxygen density of network, higher values of which correspond to higher \( T_g \). The higher the activation energy for glass transition, the more stable the structure of the glass. In a word, the results show that the thermostability performance of B glass with more Al₂O₃ addition is higher than A glass.

![DTA thermograms for (a) A and (b) B glass powders at different heating rates.](image)

**Table 2** Glass transition temperature (\( T_g \)) and the crystallization temperature (\( T_c \)) for A and B glass powders recorded at different heating rates.

| Sample | \( A \) (K/min) | \( T_g \) (K) | \( T_c \) (K) |
|--------|----------------|--------------|--------------|
| 5      | 893.54         | 1035.27      |
| 10     | 903.64         | 1054.21      |
| 15     | 911.19         | 1072.74      |
| 35     | 925.13         | 1107.12      |
|       | 10            | 909.51       | 1060.76      |
|       | 15            | 915          | 1068.93      |
|       | 25            | 919.14       | 1079.74      |
|       | 35            | 924.34       | 1089.34      |

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3.3 XRD for crystallization study

BaO is most likely modifying the silicate network, and then leads to form the hexagonal BaAl$_2$Si$_2$O$_8$ crystalline phase in the BCABS sealing glass. After the heat preservation at 1023 K for 0, 50, and 100 h, the XRD patterns of A and B glass show the evolution of crystalline phase in Fig. 6. With the increase of heating time, the peak of hexagonal celsian (22.37°) is gradually decreased, but that of the monoclinic celsian (28.65°) is increased. The result indicates that through the heat treatment, the hexagonal BaAl$_2$Si$_2$O$_8$ with the TEC of 8×10$^{-6}$ K$^{-1}$ is transformed into monoclinic BaAl$_2$Si$_2$O$_8$ (2.3×10$^{-6}$ K$^{-1}$) [29]. In Fig. 6(a), after the heat treatment at 1023 K for 50 h, the intensity of the peaks of hexagonal BaAl$_2$Si$_2$O$_8$ phase is decreased, but that of monoclinic BaAl$_2$Si$_2$O$_8$ phase is increased. A new phase (Ba$_2$Ca(B$_3$O$_6$)$_2$) at 29.17° is also appeared and unchanged with the increase of heat-treating time. However, by comparing Fig. 6(a) with Fig. 6(b), the obvious difference is that the curve in Fig. 6(b) is not changed after heating at 1023 K for 50 h, and the peak of the monoclinic BaAl$_2$Si$_2$O$_8$ phase is weaker than that of Fig. 6(a) after heating at 1023 K for 100 h. There are small humps in the XRD patterns as Fig. 6(a) and Fig. 6(b) show, which indicates that the amorphous content in glass is still present. This amorphous content is consumed by crystalline phase BaAl$_2$Si$_2$O$_8$ and Ba$_2$Ca(B$_3$O$_6$)$_2$. The above results show that through the same heat treatment, the crystalline transformation rate of A glass is faster than that of B glass, and then determin that adding Al$_2$O$_3$ could effectively delay the transformation of crystalline phase in glass, which is consistent with the analysis results of the activation energy of the glass.

### Table 3 Activation energies for the glass transition ($E_t$) and crystallization ($E_c$) of A and B glass

| Sample | ln($T_p^2/\alpha$) versus 1000/$T_p$ ($E_t$/kJ/mol) | ln($T_c^2/\alpha$) versus 1000/$T_c$ ($E_c$/kJ/mol) | ln(1/$\alpha$) versus 1000/$T_g$ ($E_t$/kJ/mol) |
|--------|---------------------------------|---------------------------------|---------------------------------|
| A      | 233.89                          | 396.99                          | 411.93                          |
| B      | 406.03                          | 594.03                          | 609.22                          |
3.4 Dilatometry measurements

To achieve the suitable sealing glass for IT-SOFCs, the TEC of the glass materials as the main criterion must be in the range of $9 \times 10^{-6}$ to $12 \times 10^{-6}$ K$^{-1}$ under working temperature [30–32]. The measured linear TECs of the two kinds of glass are shown in Fig. 7, which could determine that the glass transition points are 858 and 834 K for A and B glass, respectively. The softening points for two kinds of glass are 911 and 1035 K for A and B glass, respectively. The results show the TECs of A and B glass are approximately equal and the values are $(10.5 \pm 0.2) \times 10^{-6}$ K$^{-1}$ before the heat preservation under rigid conditions. After the heat treatment at 1023 K for 100 h, the TEC value of A glass at 1023 K for 100 h is $8.2 \times 10^{-6}$ K$^{-1}$, lower than $10.02 \times 10^{-6}$ K$^{-1}$ of B glass. It means that the crystal transformation rate in the A glass is faster than that of B glass, which is corresponding to the results of $E_c$. This phenomenon shows that the alumina could effectively increase the softening temperature of glass, which also indicates that B glass with more Al$_2$O$_3$ addition is more suitable for sealing at high temperature condition.

3.5 Interfacial microstructures

In order to further verify the better sealing performance of B glass than that of A glass, the cross-sectional morphology of two kinds of glass after heat treatment at 1123 K for 30 min followed by soaking at 1023 K for 100 h were characterized by SEM testing. Figures 8(a) and 8(c) are SEM images with a magnification of...
40 that show the sealing situation of A and B glass, respectively. Both the upper layer and the lower layer are the glass, and the middle layer is half-cell. As shown in Figs. 8(a) and 8(c), there are several cracks through the half-cell and A glass, but no cracks through the half-cell and B glass, which indicates that A glass has worse sealing performance than B glass during the long-term heat treatment. Figures 8(b) and 8(d) with a magnification of 400 are separately corresponding to the red rectangular area marked in Fig. 8(a) and Fig. 8(c). In Fig. 8(b), it could be seen that there is a clear crack through the anode and glass, which is due to the mismatch of TEC between the glass and the anode caused by the high-temperature cycling in Fig. 7. In Fig. 8(d), although the glass has been sintered relatively dense, there are still some closed pores filled in the glass region in Fig. 8(c). The appearance of closed porosity usually does not result in the enhancement of leak rate [33]. Therefore, this phenomenon shows that B glass could adhere well to half-cell and there are no cracks in the interface after 100 h heat treatment at 1073 K, which indicates that B glass is more suitable than A glass for IT-SOFCs sealing.

4 Conclusions

In this paper, the effects of Al₂O₃ addition on the thermal stability of BCABS glass were studied from the aspect of the crystallization kinetics. The FTIR spectra of glass with different Al₂O₃ content showed Al³⁺ ion is 4-coordinated and participated as network forming. XRD patterns verified that more Al₂O₃ content could decrease the formation of the monoclinic BaAl₂Si₂O₈ and Ba₂Ca(B₂O₆)₂ crystalline phase during the heat treatment at 1023 K for 100 h. Both of the activation energy for the glass transition and crystallization were increased, which effectively suppressed the crystallization ability of B glass with higher Al₂O₃ content under the heat treatment. The TEC of B glass was merely changed from (10.5±0.2)×10⁻⁶ to 10.02×10⁻⁶ K⁻¹ through the high-temperature process. The sealing performance of glass by SEM testing further confirmed that B glass with more Al₂O₃ addition could adhere well to half-cell with no cracks in the interface after heat treatment, which is more beneficial for IT-SOFCs applications than A glass.

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