



Toward excellent oxidation resistance of Al₂O₃-SiC-C castables: new insights based on a novel pore-filling agent

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Abstract

There are a lot of factors affecting the oxidation resistance of Al₂O₃-SiC-C castables. The density of the castables plays a key role and influences the Al₂O₃-SiC-C castables' physical properties, oxidation resistance, thermal shock resistance, and corrosion resistance. The aim of this work is to use spodumene as an additive to improve the oxidation resistance of the Al₂O₃-SiC-C castables. The results showed that at high-temperature, solid phases of spodumene transformed into liquid phases, which filled the pores in the Al₂O₃-SiC-C matrix. Therefore, the silicon carbide, carbon source, and micro silicon in the matrix were protected from oxidation, which promoted the generation of the silicon carbide and mullite whiskers in the matrix during sintering processes. In addition, the mechanical strength, thermal shock resistance, oxidation resistance, and corrosion resistance of specimens were improved with an increase in the content of spodumene. When the content of spodumene reached 2.0 wt.%, the specimens exhibited excellent properties. However, due to the volume expansion of the α -spodumene phase transformation, with a further increase in the content of spodumene, the properties of specimens degraded sharply. Therefore, an optimum content ~ 2.0 wt.% of spodumene was regarded as an improvement agent additive in the Al₂O₃-SiC-C castables system.

Keywords Al₂O₃-SiC-C castables · Oxidation resistance · Spodumene · Corrosion resistance · Thermal shock resistance

Introduction

Al₂O₃-SiC-C castables are popularly applied in the ironmaking system owing to their outstanding properties such as high mechanical strength, good corrosion resistance, and excellent thermal shock resistance [1–3]. Nevertheless, the oxidation resistance of Al₂O₃-SiC-C castables is poor, resulting in affecting the usability of the Al₂O₃-SiC-C castables seriously [4].

Generally, on the one hand, the oxidation resistance property of specimens is improved by reducing the porosity, which inhibits the influx of air or oxygen into the matrix. On the other hand, by adding the antioxidants including Al and Si, therefore, a few researchers have paid attention to developing methods to reduce the porosity of Al₂O₃-SiC-C castables and improving their oxidation resistance. For example, Ding et al. [5] added andalusite into Al₂O₃-SiC-C castables to improve the oxidation resistance of Al₂O₃-SiC-C castables because of the mullitization of andalusite. Furthermore, Chen et al. [6] investigated the effect of the grades of andalusite on the oxidation resistance of Al₂O₃-SiC-C castables. The results showed that the Al₂O₃-SiC-C castables with low grades of andalusite exhibited better oxidation resistance. Because at high temperatures, more liquid phases were generated, which effectively filled into pores in the matrix. Li et al. [7] adopted graphitic carbon spheres as the carbon source to prepare Al₂O₃-SiC-C castables. Owing to the good flow ability of graphitic carbon spheres, the matrix became dense, which improved the oxidation resistance of Al₂O₃-SiC-C castables. Besides, Xia et al. [8] introduced surface-treated

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Table 1 The chemical compositions of Al_2O_3 -SiC-C castables (wt.%)

Chemical composition	Li_2O	Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	IL
Mass percentage	3.50	20.50	60.21	0.12	0.87	0.02	4.20	3.10	0.07	5.1

Table 2 The formulation of Al_2O_3 -SiC-C castables/wt%

Material	0 wt.%	0.5 wt.%	1.0 wt.%	1.5 wt.%	2.0 wt.%	2.5 wt.%
Corundum aggregates (8–5, 5–3, 3–1, and 1–0 mm)	56	56	56	56	56	56
Corundum powders ($d_{50} = 74 \mu\text{m}$)	10	9.5	9	8.5	8	7.5
Alumina powders ($d_{50} = 3 \mu\text{m}$)	8	8	8	8	8	8
Silicon carbides	16	16	16	16	16	16
Micro silicon	2	2	2	2	2	2
Micro aluminum	0.1	0.1	0.1	0.1	0.1	0.1
Calcium aluminate cement	5	5	5	5	5	5
Spodumene ($d_{50} = 74 \mu\text{m}$)	0	0.5	1.0	1.5	2.0	2.5
Spherical coal tar pitch	3	3	3	3	3	3
FS 20	0.1	0.1	0.1	0.1	0.1	0.1
Sodium hexametaphosphate	0.15	0.15	0.15	0.15	0.15	0.15
Water	± 4.8	± 4.8	± 4.8	± 4.8	± 4.8	± 4.8

composite metal powders into the Al_2O_3 -SiC-C castables. Their oxidation resistance was enhanced because the exposed Al and Si react preferentially with oxygen, protecting the carbon. All above researches indeed improved the properties of the Al_2O_3 -SiC-C castables, while there existing some disadvantages of high cost, complex preparation process.

Spodumene is considered an important material for the battery industry. However, spodumene with a low content of Li loses its application value in the battery field. Normally, spodumene with a low content of Li is used as a lubricant and fluxing agent in industries [9–11]. However, few papers reported the use of spodumene to improve

the properties of refractories. Owing to the low-melting temperature of spodumene, the solid phase of spodumene transforms into the liquid phase easily at high temperature. As the treating temperature increases, the viscosity of the liquid phase decreased. Lao et al. [12] optimized the structure of pores in the reticulated porous ceramics and improved the mechanical properties of the porous ceramics by adding spodumene. But to the best of author's knowledge, no reports have detailed studied the effect of spodumene on the oxidation resistance of Al_2O_3 -SiC-C castables. Therefore, in this paper, spodumene was used

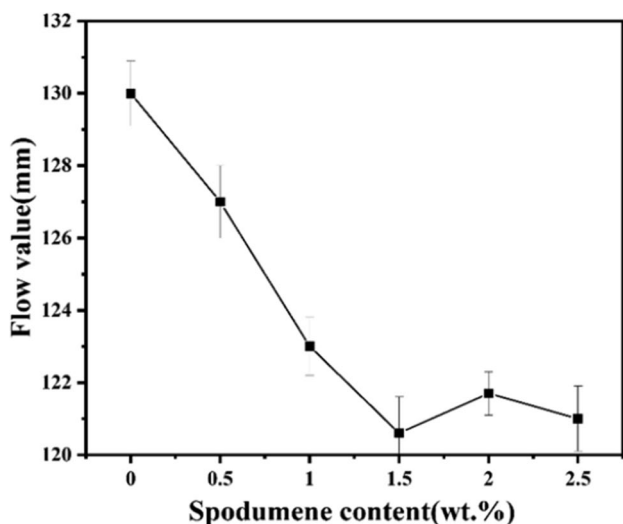
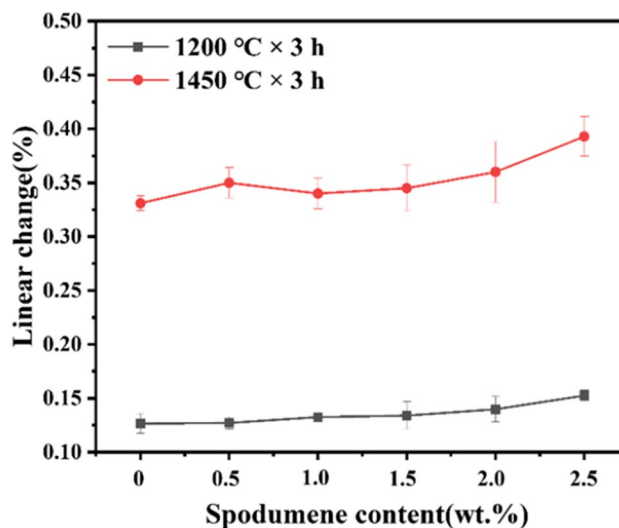
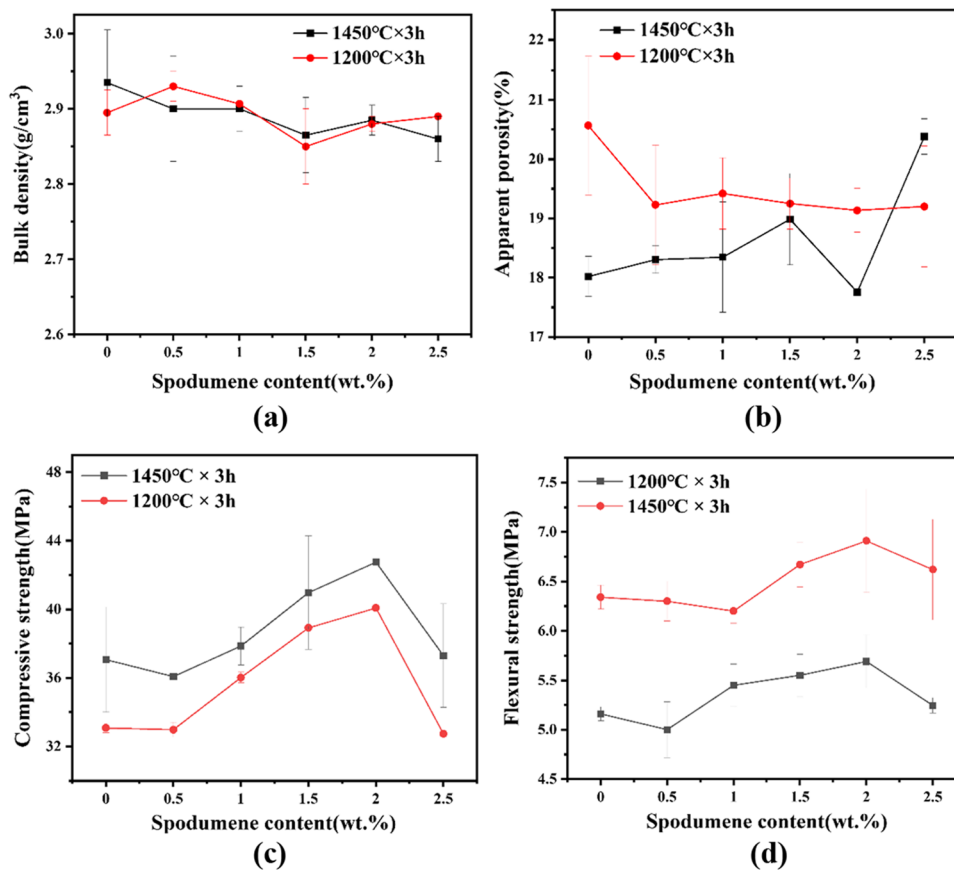
**Fig. 1** The flow value of the Al_2O_3 -SiC-C castables**Fig. 2** The linear shrinkage of Al_2O_3 -SiC-C castables treated at different temperature

Fig. 3 The mechanical properties of $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables sintered at different temperature (a) bulk density and (b) apparent porosity, (c) compressive strength, and (d) flexural strength



as a pore-filling agent to prepare the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables. Furthermore, the effects of the spodumene content on the oxidation resistance, corrosion resistance, thermal shock resistance, high-temperature strength, and the physical properties of $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables were investigated.

Experiment section

Materials

Corundum aggregates ($\text{Al}_2\text{O}_3 \geq 97$ wt.%, 8–5 mm, 5–3 mm, 3–1 mm, and 1–0 mm, Xinrui Co., Ltd., China), silicon carbide powder ($\text{SiC} \geq 98$ wt.%, ≤ 1 mm, Ningxia Hexing Carbon-Based Materials Co., Ltd., China), alumina powder ($\text{Al}_2\text{O}_3 \geq 98$ wt.%, $d_{50} = 74$ μm and $d_{50} = 3$ μm , Shandong Hengjia High Purity Aluminum Technology Co., Ltd., China), micro silicon powder ($\text{SiO}_2 \geq 95$ wt.%, $1 \leq \mu\text{m}$, Elkem Microsilica

915U), calcium aluminate cement (Secar 71, Lafarge Group, China), spodumene powder (Li content ≤ 5 wt.%, ≤ 0.074 μm , Linxiang Huachang Nonmetallic Ore Co., Ltd., China), and spherical coal tar pitch (≤ 0.5 mm, Zhenjiang Xinguang New Material Technology Co., Ltd., China) were used as raw materials. Polycarboxylate (FS20, BASF Co., Ltd., German) and sodium hexametaphosphate (Chongqing Chuandong Chemical Co., Ltd.) were regarded as dispersants. The chemical compositions of spodumene are listed in Table 1. The formulations of $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables are listed in Table 2.

Experimental setup and method

The various materials are weighted according to Table 1 and mixed uniformly. The mixture was cast into $40 \times 40 \times 160$ mm bars under vibration and then dried at 110 $^\circ\text{C}$ for 24 h. Finally, the $\text{Al}_2\text{O}_3\text{-SiC-C}$ green bodies were treated at 1450 $^\circ\text{C}$ for 3 h to prepare $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens.

Table 3 The viscosity of liquid phase of spodumene at different temperature as calculated by the FactSage viscosity module (log(poise))

Temperature	1200 $^\circ\text{C}$	1250 $^\circ\text{C}$	1300 $^\circ\text{C}$	1350 $^\circ\text{C}$	1400 $^\circ\text{C}$	1450 $^\circ\text{C}$
Spodumene	6.807	6.289	5.809	5.364	4.952	4.570

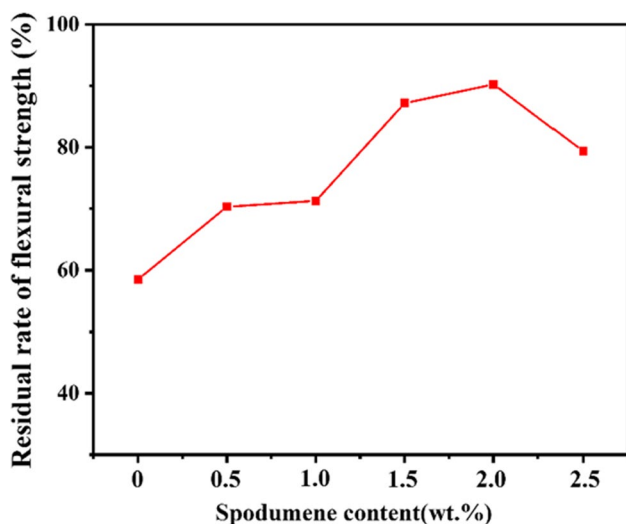


Fig. 4 The residual rate of flexural strength $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables after thermal shock testing for five cycles

Characterization techniques

The flow ability of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables was characterized based on GB/T 2419-2005. The linear change of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables was characterized by the width of specimens measured before and after sintering. The porosity and density of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens were determined using the Archimedes methods. The cold compressive strength of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens was tested using a

universal testing machine (Shenzhen Wance Test Equipment Co., Ltd.) with a ram speed of 0.5 mm/min. The thermal shock resistance of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens was evaluated using the water quenching technique at 1100 °C for 20 min in air and then immersed in water for 5 min. The modulus of rupture after 5 cycles of thermal shock (MORst) was measured and the residual strength ratios were calculated. Residual strength ratio = $(\text{MORst}/\text{CMOR}) \times 100\%$ [13]. The alkali corrosion resistance of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens was measured according to the Chinese National Standard GB/T 14983-2008 [14]. The oxidation resistance degree of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens was observed from its oxidized layer area on the fracture surface of the specimens, and the oxidation resistance index (%) of specimens was measured using the Image-Pro Plus software [15]. The micromorphology of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ specimens was characterized by scanning electron microscopy (SEM, ISM-6610, JEOL, Japan). The viscosity of the melting liquid phase was studied via using the viscosity module of FactSage software.

Results and discussion

Figure 1 shows the flow values of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables with different content of spodumene. As the content of spodumene increased, the flow value of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables decreased gradually. In this work, the density of corundum was larger than that of spodumene. Therefore, the number of spodumene powder is more than that of

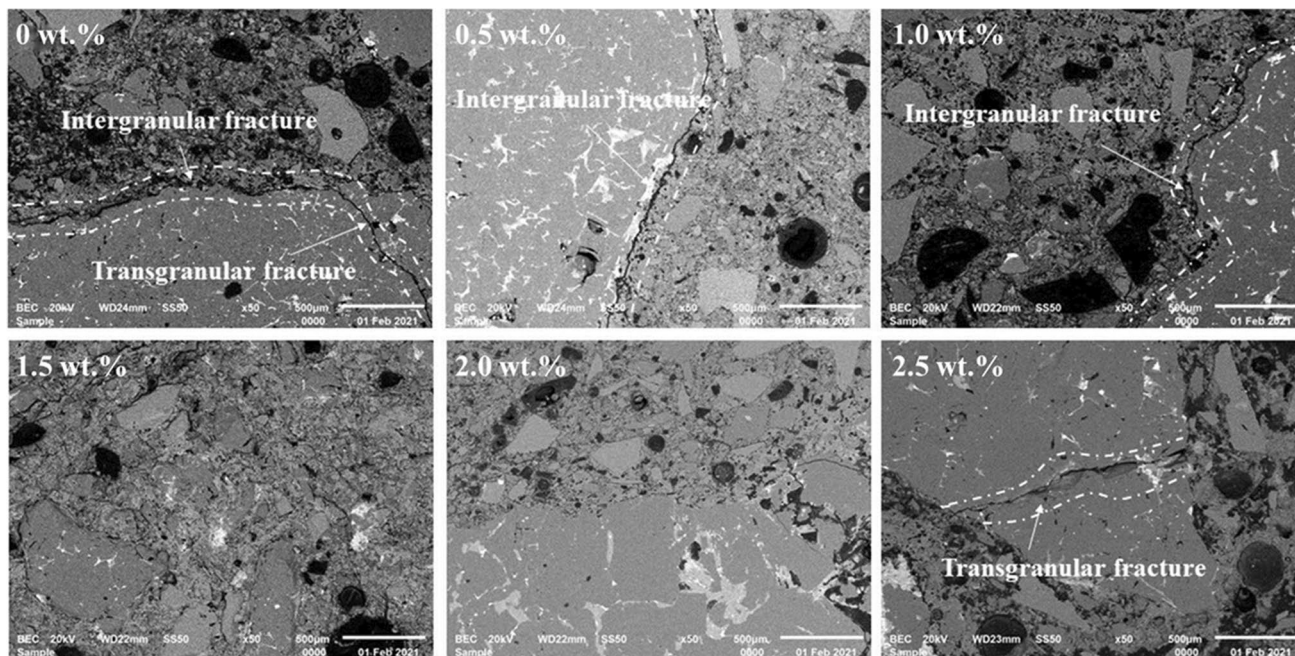


Fig. 5 The microstructure of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables after thermal shock testing for five cycles

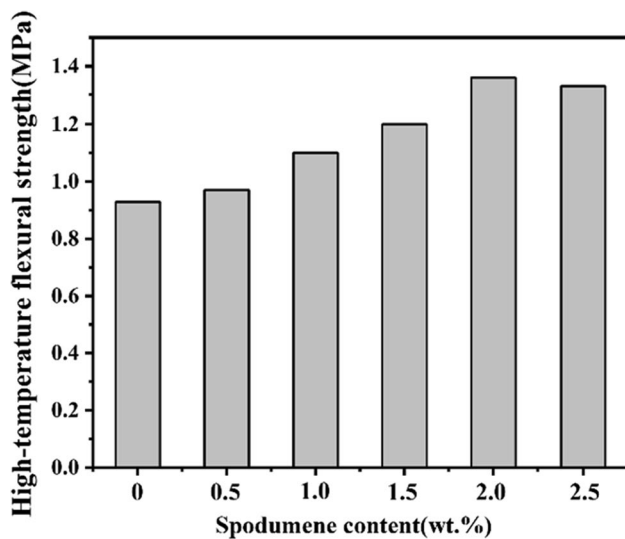


Fig. 6 The high-temperature flexural strength Al₂O₃-SiC-C castables

corundum powder under the same quality conditions, which affected the flow ability of the Al₂O₃-SiC-C castables under the same amounts of water addition [16].

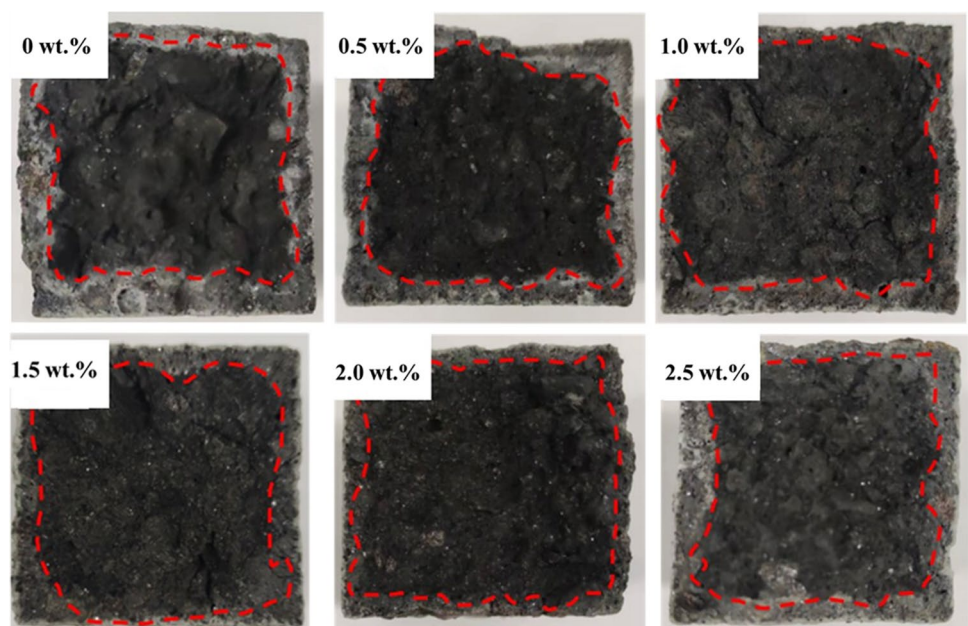
Figure 2 shows the linear change of the different Al₂O₃-SiC-C castables treated at 1200 °C and 1450 °C. When the temperature was 1200 °C, with an increase in the content of spodumene, the linear change of specimens increased. Compared to the specimens treated at 1200 °C, the linear change of the specimens treated at 1450 °C increased sharply. Because a large amount of liquid phases (spodumene) with low viscosity were generated at 1450 °C, which improved the sintering process and filled pores, the bulk density and apparent porosity of the specimens

decreased gradually with an increase in the content of spodumene (Fig. 3). Because at high-temperature liquid phases were generated, which promoted the sintering process of the specimens [17], meanwhile, at high temperature, the viscosity of the generated liquid phase decreased significantly, which was beneficial to reduce the porosity of the specimens. The generated liquid phase effectively promoted the formation of silicon carbide and mullite whiskers, resulting in filling the pores.

Compared to the physical properties of the specimens without spodumene, the specimens with spodumene showed excellent flexural strength and compressive strength (Fig. 3). As shown in Fig. 3a and b, with the spodumene increased, the bulky density and the porosity both decreased, and the reason might be attributed to that a lot of closed pores in the matrix, which led to reducing the density of the specimens. Moreover, the compressive strength and flexural strength of the specimens increased with the contents of spodumene. The generation of mullite and SiC fibers was promoted by the formation of the liquid phase with low viscosity at high temperature (Table 3) [18]. Generally, at high temperature, the liquid phases filling into the pores inhibit the oxidation of carbon source and silicon. Moreover, the liquid phase with high fluidity accelerated the transport of the substances in the matrix. When the content of spodumene was 2.0 wt.%, the strength of the specimens reached the maximum. With a further increase in the content of spodumene, the strength of the specimens decreased, due to the volume expansion of the phase transformation of spodumene and the abnormal grain growth.

Figure 4 depicts the residual ratio of the flexural strength of specimens. The residual rate of the specimens increased with the content of spodumene. When the content of

Fig. 7 The cross sections of the Al₂O₃-SiC-C castables heated at 1450 °C for 3 h in air



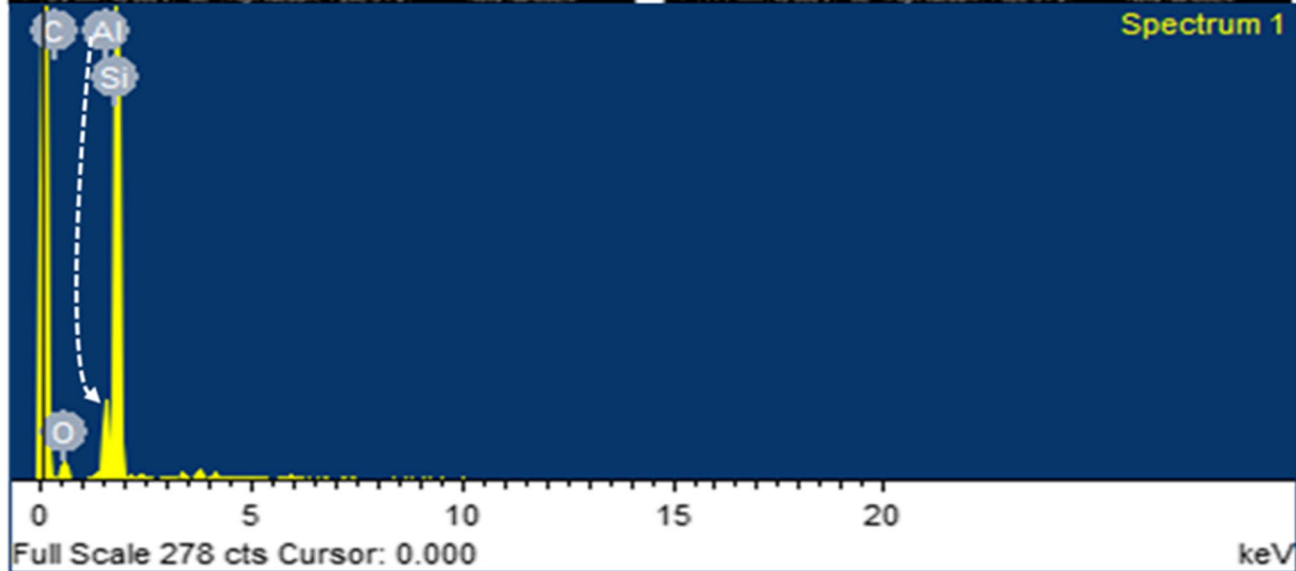
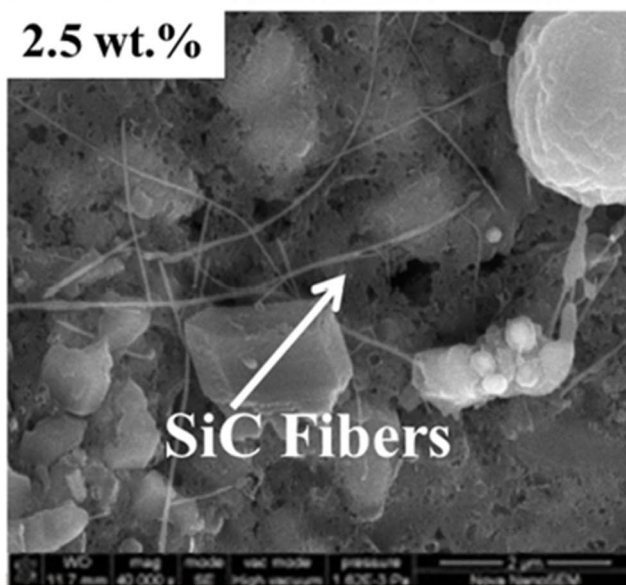
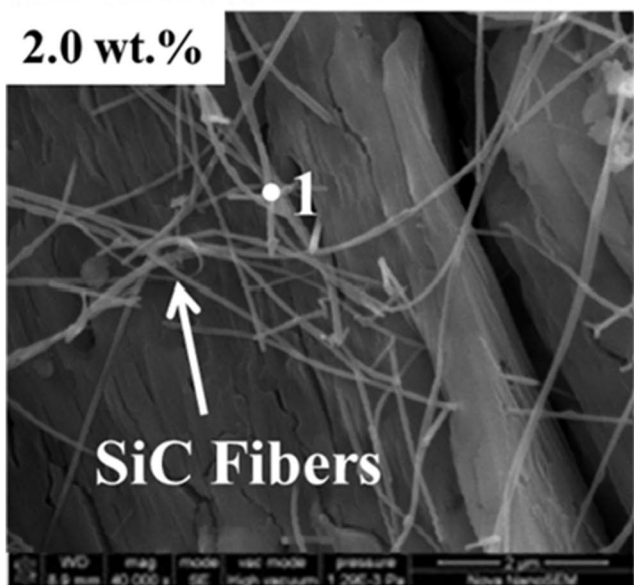
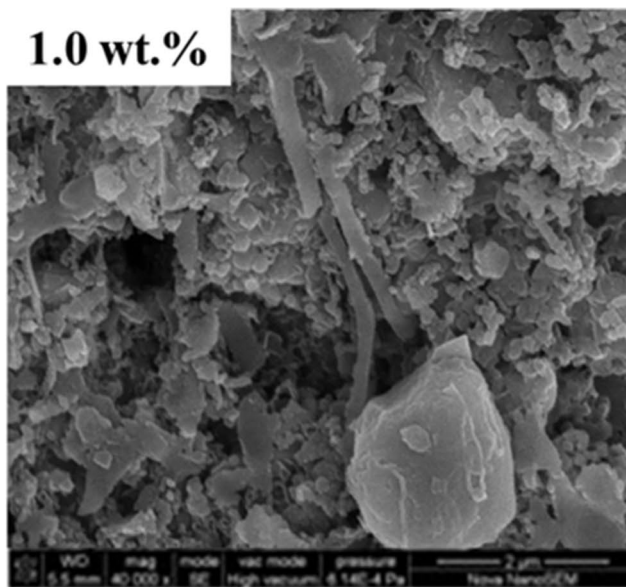
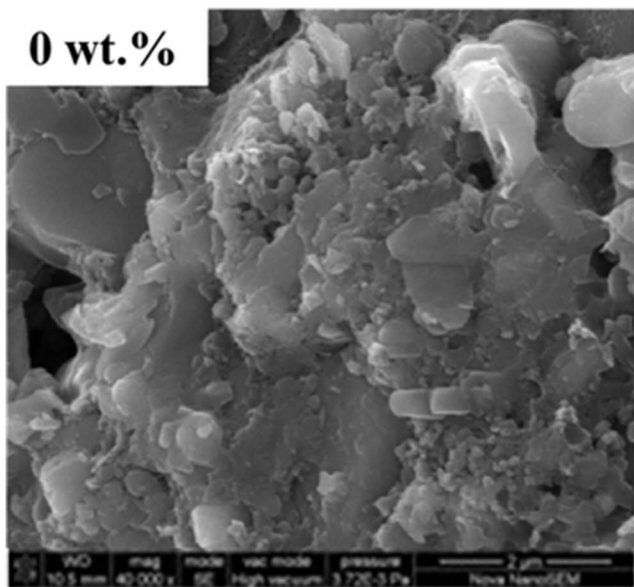


Fig. 8 The microstructure of the unoxidized parts of the Al_2O_3 -SiC-C castables heated at 1450 °C for 3 h and EDS results of the sample with 2.0 wt% spodumene

spodumene was 2.0 wt.%, the residual ratio of flexural strength reached the maximum, which indicated that the specimens with 2.0 wt.% spodumene owned the outstanding thermal shock resistance. It was beneficial to prevent the oxidation of SiC and C, adding spodumene powders into the Al_2O_3 -SiC-C matrix, owing to low the porosity of specimens. Moreover, SiC whiskers were generated in the matrix, which was helpful to improve the thermal shock resistance of the specimens. Hasselman's theory was also used to predict the thermal shock resistance (Eqs. (1) and (2)) [19, 20]

$$R = \sigma(1 - \nu)/\alpha E \quad (1)$$

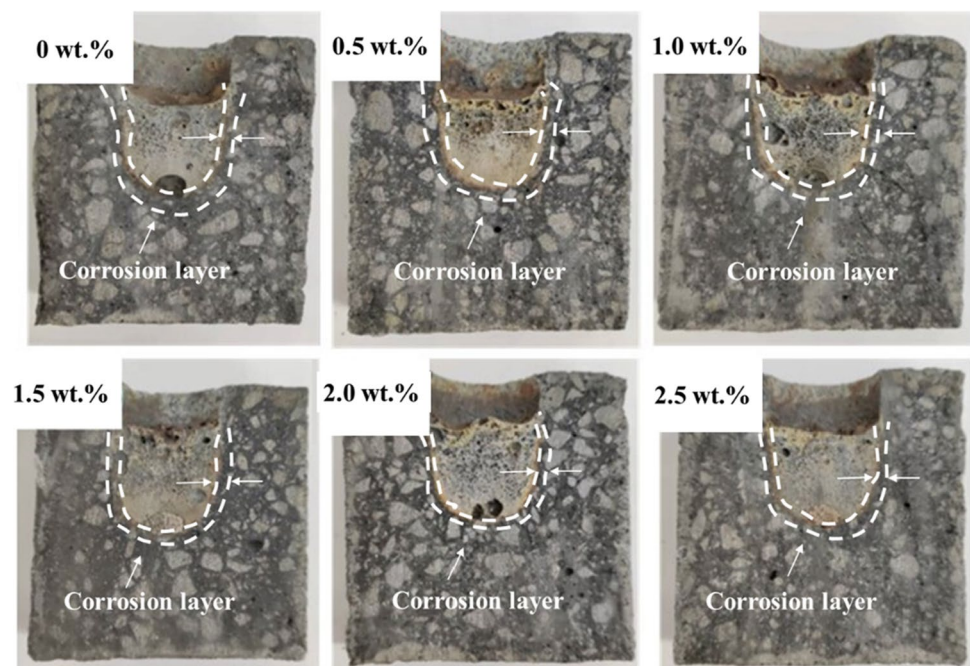
where R is a measure of the resistance to crack initiation, σ is the strength, ν is the Poisson ratio, α is the thermal expansion coefficient, and E is Young's modulus. The Young's modulus the specimens were decreased by adding spodumene because of high porosity of the specimens with spodumene, which had a positive influence on the improvement of thermal shock resistance of the specimens [21]. However, when an excess content of spodumene ~ 2.5 wt.% was added into the matrix, a huge amount of liquid phases were generated, which were harmful to the thermal shock resistance of the specimens.

Figure 5 presents the microstructure of the Al_2O_3 -SiC-C castables after thermal shock testing. It could be seen that

the number of pores in the matrix was reduced significantly by adding spodumene. As the content of spodumene increased, the size of the pores also decreased, which was beneficial to improving the strength and thermal shock resistance of the specimens. There are few cracks and flaws in the specimens with 2.0 wt.% spodumene. But the large cracks were formed in the specimens with 0, 0.5, 1.0, and 2.5 wt.% content of spodumene. For the specimens with a low content of spodumene, the liquid phases were not enough to fill the most of pores. Thus, a lot of large pores were still in the specimens, and few silicon carbide and mullite whiskers were generated in the matrix, which led to the poor thermal shock resistance of the specimens. For the specimens with the excess content of spodumene, a large amount of liquid phases with low viscosity generated in the matrix and the volume expansion of phase transformation of α -spodumene influenced the thermal shock resistance of the specimens [22].

The high-temperature flexural strength of the Al_2O_3 -SiC-C castables is shown in Fig. 6. It was beneficial to improve the high-temperature flexural strength by adding spodumene. The liquid phase generated by spodumene has a positive influence on the density of specimens and the growth of the mullite phase in the matrix. In addition, the carbon source, silicon carbide, and micro silicon were prevented from oxidizing by adding spodumene. Therefore, at high temperature, the carbon source- spherical coal tar pitch reacted with micro silicon to form SiC whiskers, which was helpful for the improvement of the high-temperature strength.

Fig. 9 The fracture surface of Al_2O_3 -SiC-C castables after corrosion



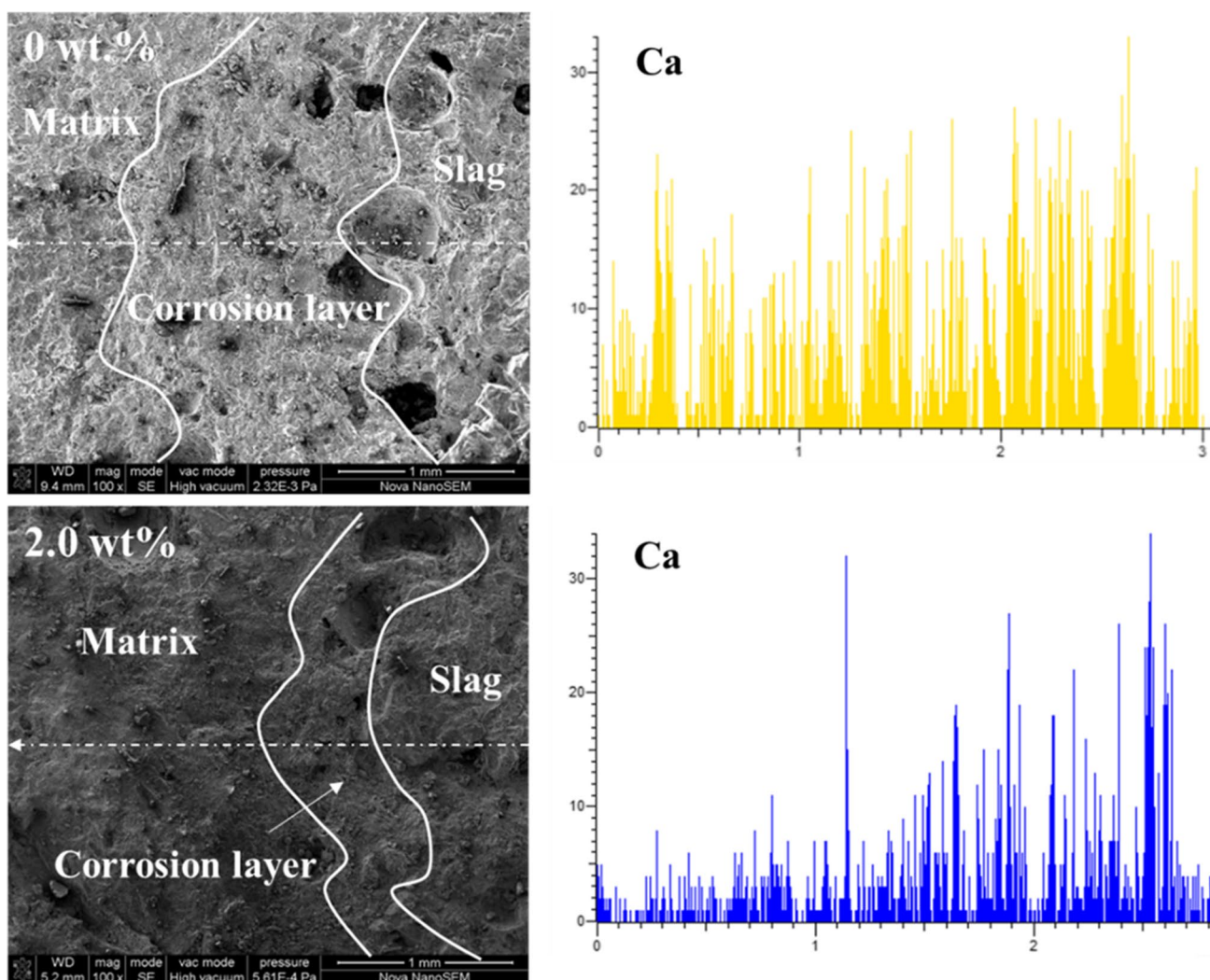


Fig. 10 The microstructure of the specimens with 0 wt% and 2.0 wt% after corrosion testing

Figure 7 shows the morphology of fracture surfaces of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ castables heated at $1450\text{ }^\circ\text{C}$ for 3 h in air. As the content of spodumene increased, the thickness of the oxide layer decreased gradually. When the content of spodumene reached 2 wt.%, the specimens showed the best oxidation resistance. With a further increase in the content of spodumene, the oxidation resistance of specimens decreased sharply. The softening temperature of spodumene $\sim 1248\text{ }^\circ\text{C}$ was lower than the sintering temperature of the $\text{Al}_2\text{O}_3\text{-SiC-C}$ matrix [23]. The α -spodumene transformed into β -spodumene at $1082\text{ }^\circ\text{C}$, which caused volume expansion about $\sim 30\%$. Therefore, when the excess amount of spodumene was added to the matrix, the matrix would expand significantly [22]. Thus, the number of cracks and flaws generated in the specimens with 2.5 wt.% spodumene, and then air filled the matrix easily through the cracks and flaws, resulting in the poor oxidation resistance of the specimens.

In addition, as the pores in the matrix were filled with melting liquid phase, air was effectively prevented from contacting the spherical coal tar pitch, micro silicon, and silicon carbide in the matrix [24]. Therefore, an unoxidized carbon source (spherical coal tar pitch) reacts with micro silicon to form silicon carbide fibers as shown in Fig. 8. The EDS results indicated that the fibers were SiC; moreover, with the spodumene amounts increased, the SiC fibers were formed, the number of silicon carbide fibers increased, and the length diameter ratio also increased gradually, which might be attributed to the generated liquid phase promoted the generation of silicon carbide fibers [25]. However, when the content of spodumene reached 2.5 wt.%, the pores and cracks were formed in the matrix due to the volume expansion of the α -spodumene phase transformation, which caused the spherical coal tar pitch

and micro silicon to be oxidized. Therefore, compared to the specimens with 2.0 wt.% content of spodumene, the number of silicon carbide fibers and the diameter of silicon carbide fibers in the specimens with 2.5 wt.% decreased significantly. Thus, when the content of spodumene was higher than 2.0 wt.%, the mechanical strength of the specimens decreased as the content of the spodumene increased.

Figure 9 shows the micromorphology of Al_2O_3 -SiC-C castables with the different content of spodumene after slag corrosion at high temperature. It was observed that thin corrosion layers were formed in the specimens. The thickness of the corrosion layer in the specimen with 0.2 wt.% was thinner than that in other specimens (Figs. 9 and 10). Compared to the specimens without spodumene, the porosity of specimens with spodumene reduced obviously, which effectively prevented the melting slag from penetrating the matrix. Moreover, the silicon carbide whiskers were generated in the matrix. Owing to the poor wettability between silicon carbide/spherical coal tar pitch and slags, as well the low porosity of the specimens, the slag corrosion resistance of the castables with spodumene was improved.

Conclusion

In this work, the effect of spodumene on the properties of Al_2O_3 -SiC-C castables was investigated. At high temperature, the solid phase of spodumene transformed into the liquid phase. Therefore, using spodumene as pore filler not only reduced the porosity in the matrix but also promoted the generation of mullite and SiC whiskers in the matrix, which had a positive influence on the oxidation resistance and thermal shock resistance properties of Al_2O_3 -SiC-C castables. When the content of spodumene reached 2.0 wt.%, the specimens owned the best oxidation resistance. However, with a further increase in the amount of spodumene, the properties of specimens decreased due to the volume expansion caused by the phase transformation of α -spodumene and a large amount of liquid phase. Therefore, it is an effective method to prepare the Al_2O_3 -SiC-C castables with good oxidation resistance by adding an optimum addition amount of spodumene ~ 2.0 wt.%.

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Declarations

Conflict of interest The authors declare no competing interests.

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