

Enhancement of bonding network for silica sol bonded SiC castables by reactive micropowder



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ABSTRACT

Silica sol bonded castables have obvious advantages over low cement or hydratable alumina bonded castables in drying performance and sintering properties for SiC castables. However, they are not widely used due to their weak strength at low temperature. The efficiency of bonding network for silica sol bonded SiC castable in the presence of different reactive micropowder such as SiO₂ micropowder and α-Al₂O₃ micropowder was evaluated through oscillatory tests, sintered properties and microstructural analysis. Results show that the polymerization reaction between SiO₂ micropowders enhanced the siloxane network and reinforced the bonding strength, furthermore, the addition of α-Al₂O₃ micropowder contributed to accelerating the formation of the siloxane network and hardening of the silica sol at lower temperatures and shorter time. Silica sol performed well as a binder agent for SiC castables with an addition content of 3 wt% SiO₂ micropowder and 2 wt% α-Al₂O₃ micropowder, which showed high strength and good workability at room temperature. And Silica sol bonded SiC castable with the above micropowder contents possessed the best mechanical behavior after heat treatment due to combined binding of SiC whiskers and mullite.

1. Introduction

With rapid economic development, the amount of municipal waste has risen sharply. Presently, nearly one billion tons of waste is produced per year in China, which causes serious pollution of water, soil, and atmosphere. The high-temperature solid waste gasifier possesses great advantages in dealing with solid waste, particularly hazardous wastes [1] (medical waste, electronic waste, etc.) as it avoids the formation of noxious chemicals in the ash such as heavy metals [2,3], PAHs [4], and dioxins like PCDD/PCDFs [5,6] due to its high operating temperature (over 1500 °C around the torch). However, higher operating temperature creates strict requirements for the lining refractory of the gasifier. Excellent mechanical properties at high temperature indicate that the silica sol bonded SiC castable has prospects for application in the high-temperature solid waste gasifier [7].

Calcium aluminate cement (CAC) is the most commonly used hydraulic binder in refractory castables. However, the presence of CaO in the composition can be deleterious to the refractories of some ceramic systems, such as those containing microsilica. Silica sol particles can be linked together using different setting mechanisms

such as gelling and coagulation, providing initial strength when applied to refractories. The interaction of silica particles triggers siloxane bonding (Si–O–Si) and the building of a three-dimensional network. The change of chemical bonding provides strength without the formation of hydration products, which can avoid the cracking caused by water vapor jammed by hydration products during drying.

Nevertheless, silica sol bonded castables are still not widely used due to their inferior strength at low temperature and long setting time. MgO addition in the range of 0.3–0.6 wt% could increase the green strength of silica sol bonded castables [8] through bond formation (–Si–O–Mg–O–Si–) to accelerate the three-dimensional network [9]. Some reports in the literature considered the use of CAC as a setting agent for silica sol [10]. However, the presence of CaO in the composition may be deleterious to the refractories. Zhang [11] thought that the addition of MgO and CAC benefit the siloxane network formation of silica sol bonded Al₂O₃–SiC–C castables but harm the workability. SiO₂ micropowder and α-Al₂O₃ micropowder are widely used in the matrix of current refractory castable formulations. In refractory castables, SiO₂ micropowder fills the voids initially occupied by water, improving system packing. When alumina is present, it also induces mullite formation, increasing the hot-strength. In this paper, the effects of

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addition of alumina micropowder and silica micropowder on the mechanical behavior of a silica sol bonded SiC castable were studied. The sintering and mechanical properties after heat treatment at different temperatures (110 °C and 1500 °C) of silica sol bonded castable were determined. To obtain a better understanding of the interactions, a systemic rheological approach using oscillatory tests to evaluate the coagulation behavior was carried out. Oscillatory tests make it possible to differentiate the elastic and viscous responses, which are expressed by the storage modulus (G') and the loss modulus (G''), respectively. G' represents the elastic component and G'' the viscous component. The normal force (N_f) shows the specimen response to an elastic deformation imposed uniaxially [12]. Both tests characterize the viscoelastic properties of materials, which can be related to the physical changes during the formation of the three-dimensional siloxane network ($-\text{Si}-\text{O}-\text{Si}-$) as the silica sol changes from a low viscous liquid to an elastic solid. The evaluation of refractory suspensions using viscoelastic properties (G' and N_f) explained the coagulation behavior of the silica sol bonded SiC castable.

2. Experimental

A kind of silica sol bonded SiC castable was prepared. SiC was chosen as the aggregate. SiC aggregates, SiC powder (≤ 0.088 mm), Si powder (≤ 0.088 mm), carbon black (≤ 0.045 mm), $\alpha\text{-Al}_2\text{O}_3$ micropowder (D_{50} 2 μm), SiO_2 micropowder (D_{50} 0.775 μm) and silica sol (30 wt % of SiO_2) were weighed, mixed, and cast as $25 \times 25 \times 125$ mm blocks.

Samples with a size of $25 \times 25 \times 125$ mm were heated at 110 or 1500 °C for 3 h. The linear change rate, apparent porosity, bulk density, cold modulus of rupture (CMOR), and cold crushing strength (CCS) of the samples were measured according to ISO 2477:2005, 5017:1998, 5014:1997, and 8895:2004, respectively.

Slurries prepared by mixing SiC aggregates (1–0 mm), SiC powder (≤ 0.088 mm), Si powder (≤ 0.088 mm), carbon black (≤ 0.045 mm), $\alpha\text{-Al}_2\text{O}_3$ powder (D_{50} 2 μm), SiO_2 micropowder (D_{50} 0.775 μm), and silica sol (30 wt% of SiO_2) was prepared for oscillatory tests through a rotary rheometer (Physica MCR301, Anton Paar, Austria) to obtain G' and N_f . The vane sensor was used in the test with a heating rate of 2 °C/min from 20 °C to 85 °C.

Based on Fig. 1, the prepared slurry was poured into the cylinder and a sinusoidal strain $\gamma(t)$ was applied on the slurry when the vane sensor oscillated with an angular frequency ω . Then the slurry gave a feedback of a sinusoidal stress $\sigma(t)$ to the rotary rheometer. Fig. 2 shows the principle of oscillatory tests, the elastic hysteresis of the slurry led to a phase lag δ between the input sinusoid $\gamma(t)$ and the output sinusoid $\sigma(t)$. For ideal fluid δ is $\pi/2$, and 0 for ideal solid. γ_0 and σ_0 are amplitudes of the sinusoids (shown in Eqs. (1) and (2)). And $\sigma(t)$ consist of the storage part and the loss part (Eq. (2)), the ratios between their amplitudes and γ_0 (shown in Eqs. (3) and (4)) were defined as the storage modulus (G') and the loss modulus (G''), respectively. G' and G'' vary with the change of the phase lag δ when silica sol changes from a low viscous liquid to an elastic solid (δ from $\pi/2$ to 0). The normal force (N_f) is the axial force of the rotary rheometer.

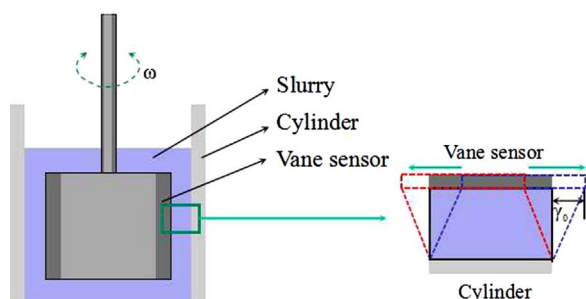


Fig. 1. Schematic drawing of oscillatory test.

G' and N_f were used to explain the coagulation behavior of the silica sol bonded SiC castable.

$$\gamma(t) = \gamma_0 \sin \omega t \quad (1)$$

$$\sigma(t) = \sigma_0 \cdot \sin(\omega t + \delta) = \sigma_0 \cdot \cos \delta \cdot \sin \omega t + \sigma_0 \cdot \sin \delta \cdot \sin(\omega t + \pi/2) \quad (2)$$

$$G' = \frac{\sigma_0 \cdot \cos \delta}{\gamma_0} \quad (3)$$

$$G'' = \frac{\sigma_0 \cdot \sin \delta}{\gamma_0} \quad (4)$$

Microstructures and morphologies of the samples were examined using a field emission scanning electron microscope (SEM, Nova NanoSem 400). The samples for SEM were coated with gold and energy-dispersive spectroscopy (EDS) was used to assist in phase identification.

3. Results and discussion

3.1. Oscillatory tests

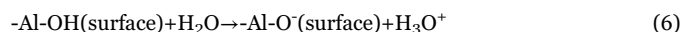
3.1.1. Oscillatory tests with different contents of SiO_2 micropowder

The influences of SiO_2 micropowder and $\alpha\text{-Al}_2\text{O}_3$ micropowder on the coagulation behavior of silica sol were taken into consideration. The slurries with different contents of SiO_2 micropowder and $\alpha\text{-Al}_2\text{O}_3$ micropowder were tested. Fig. 3(a) and (b) showed the G' and N_f of slurries with different contents of SiO_2 micropowder (2 wt% of $\alpha\text{-Al}_2\text{O}_3$ micropowder in each given slurry) at each temperature. The G' values were fitted using a polynomial of degree four, and were apparently not affected at temperatures below 50 °C. However, with the continued increase of temperature, G' and N_f increased, which indicated that the gelling and hardening behavior occurred. The slurry with more SiO_2 micropowder present had higher elasticity behavior meaning acceleration of the network formation of the silica sol. Fig. 3(c) and (d) show enlarged views of G' at temperature ranges of 20–30 °C and 40–60 °C, respectively. The former temperature range shows the initial G' values of the slurries. The slurry with 3 wt% content of SiO_2 micropowder presents a smaller G' , which could favor workability of the slurry. The G' values began transforming obviously between 50 °C and 60 °C, the change points of the samples were all about 53.6 °C (Fig. 3(d)) independent of the content of SiO_2 micropowder. Hence, the difference of N_f may be caused by the reaction among SiO_2 micropowder. The surface of SiO_2 micropowder contains Si-OH groups. The reaction of Eq. (5) occurred to improve the bonding strength [14].



3.1.2. Oscillatory tests with different contents of $\alpha\text{-Al}_2\text{O}_3$ micropowder

Samples with different contents of $\alpha\text{-Al}_2\text{O}_3$ micropowder (3 wt% of SiO_2 micropowder in each slurry) were prepared and tested. The results pointed out that G' and N_f increased with the increase of $\alpha\text{-Al}_2\text{O}_3$ micropowder; the values changed little at low temperature (Fig. 4). However, with the continued increase of temperature, G' and N_f increased. In addition, the transformation temperatures of the slurries differed with the content of $\alpha\text{-Al}_2\text{O}_3$; slurries with 0 wt%, 2 wt%, and 6 wt% $\alpha\text{-Al}_2\text{O}_3$ micropowder had transformation temperatures of 62.5 °C, 53.6 °C, and 47.8 °C (Fig. 4(d)), respectively, which indicated that $\alpha\text{-Al}_2\text{O}_3$ micropowder accelerated the siloxane network formation at lower temperature. When $\alpha\text{-Al}_2\text{O}_3$ micropowder with great activity was added to the basic silica sol, Eq. (6) [15] occurred and increased the ion concentration of the slurry.



Eq. (6) accelerated the consolidation of silica sol. And Fig. 5 shows

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