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Preparation and thermal shock resistance of cordierite-spodumene composite ceramics for solar heat transmission pipeline

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ABSTRACT

Cordierite-spodumene composite ceramics with 5, 10, 15 wt% spodumene used for solar heat transmission pipeline were in-situ prepared via pressureless sintering from kaolin, talc, γ-Al_2O_3 and spodumene. Effects of spodumene on densification, mechanical properties, thermal shock resistance, phase composition and microstructure of the composite ceramics were investigated. The results showed that spodumene used as flux material decreased the sintering temperature greatly by 40–80 °C, and improved densification and mechanical properties of the composite ceramics. Especially, sample A3 with 10 wt% spodumene additive sintered at 1380 °C exhibited the best bending strength and thermal shock resistance. The bending strengths of A3 before and after 30 thermal shock cycles (wind cooling from 1100 °C to room temperature) were 102.88 MPa and 96.29 MPa, respectively. XRD analysis indicated that the main phases of the samples before 30 thermal shock cycles were α-cordierite, α-quartz and $MgAl_2O_4$, and plenty of β-spodumene appeared after thermal shock. SEM micrographs illustrated that the submicron β-spodumene grains generated at the grain boundaries after thermal shock improved the thermal shock resistance. It is believed that the cordierite-spodumene composite ceramics can be a promising candidate material for heat transmission pipeline in the solar thermal power generation.

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1. Introduction

Concentrated solar power has been one of the most promising technologies to reduce CO_2 emissions and solve the energy crisis for its renewable energy resource and high efficiency transformation. Solar thermal power plant (STPP) is constituted by the three subsystems of central receiver, heat transfer and thermal storage [1]. And the heat transfer subsystem is the important component to transmit absorbed heat from the central receiver to the thermal storage, which directly determines the generating efficiency of the STPP. Enhancing operation temperature of the STPP has been regarded as the effective method to achieve large-scale solar power. Average outlet temperatures of central receivers have been operated over 800 °C in many solar plants, and the maximum outlet temperatures are 960 °C in the PLVCR receiver and 1200 °C in the DIAPR receiver, respectively [2]. Thereby, heat transmission pipelines in the heat transfer subsystem are required good air tightness, high mechanical strength and excellent heat resistance to guarantee the working stability of the STPP in high temperature condition. Excellent thermal stability is also a vital requirement for the pipelines materials to resist thermal shock at the temperature range of RT∼800 °C. Nowadays transmission pipelines used for fossil fuel plants and chemical plants are mostly fabricated by heat resistant steel materials [3,4]. However, the alloy steel materials have poor corrosion resistance, oxidation resistance and heat resistance. The ultimate operating temperatures of the commercial heat resistant steels such as pearlite steels, ferritic steels and austenite steels are only about 500–650 °C, and the nickel-based alloys owned better heat resistance are expensive and immaturity [5–7]. The short service life of the steel materials at high temperature made them unsuitable for high temperature applications [8,9]. Therefore, ceramics with high heat resistance, outstanding corrosion resistance and good mechanical properties have been considered as candidate materials for heat transmission pipeline in the high temperature solar thermal power.

Cordierite is magnesium alumina silicate which is widely used in high temperature applications such as refractory materials, catalyst supports, burner nozzles, heat exchanger for gas turbines and others for its low thermal expansion coefficient, high heat resistance, excellent mechanical properties and thermal shock resistance [10]. Due to very rarely obtain in nature, the majority of cordierite is synthesized from MgO-Al_2O_3-SiO_2 ternary system using the natural raw materials and synthetic powders based on magnesium, aluminum and silicon oxides [11,12]. However, the narrow sintering temperature range and incongruent melting result in the difficult preparation of dense cordierite ceramics [13].

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Cordierite ceramics prepared by pressureless sintering method usually have a high porosity. For instance, Thomaidis et al. [14] obtained the cordierite ceramic with 17.5% porosity and 1.75 g cm^{-3} density using 31 wt% bauxite, 53 wt% kaolin and 16 wt% magnesite at 1350 °C, of which had a slight deformation sintered at 1400 °C. Albhilil et al. [15] prepared the cordierite composite ceramic with 30.22% total porosity at 1400 °C using kaolin, quartz, $Al(OH)_3$ and magnesite as raw materials. In the research of Rundans et al. [16], cordierite ceramics with 1.1% apparent porosity, 2.35 g cm^{-3} bulk density and 154 MPa compressive strength were prepared by sand, $MgCO_3$ and $Al(OH)_3$. Cordierite composite ceramic prepared by Yan et al. [17] had a high apparent porosity of 44% at 1430 °C. Although other synthetic methods like sol-gel, crystallization from glasses and hot pressure have successfully fabricated high density cordierite ceramics, the complicated process and high cost prevent the large-scale production of the high density cordierite ceramics [18–20]. Sintering additives like CaO, ZnO and TiO_2 have been employed to improve the density of cordierite ceramics. Unfortunately, thermal expansion coefficient of the ceramics increased, and the thermal shock resistance reduced after these additives introducing [21–23]. Spodumene with low thermal expansion coefficient has been used as the sintering additive in Si_2N_2O ceramic, andalusite composite and other ceramics to improve thermal shock resistance [24,25]. However, few literatures have reported the method to prepare the dense cordierite ceramics with excellent thermal shock resistance using spodumene additive.

The aim of this paper is to prepare the cordierite-spodumene composite ceramics with high thermal stability and excellent properties for heat transmission pipeline in the solar thermal power plant. The dense cordierite-spodumene composite ceramics with high bending strength and excellent thermal shock resistance were in-situ synthesized by kaolin, talc, γ-Al_2O_3 and spodumene additive via pressureless sintering technology. The effects of spodumene on densification, mechanical properties, thermal shock resistance, phase composition and microstructure of the composite ceramics were studied. And the improvement mechanism of thermal shock resistance of the ceramics was analyzed.

2. Experimental procedure

Suzhou kaolin (~250 mesh, China Kaolin Co., Jiangsu, China), talc (~250 mesh, Guilin Talc Development Co., Guangxi, China), γ-Al_2O_3 (~250 mesh, Shandong Alumina Industry Co., Shandong., China) and spodumene (~250 mesh, Sichuan Tianqi Industrial Co., Sichuan, China) are employed as raw materials to prepare cordierite-spodumene composite ceramics. The chemical compositions of the raw materials performed by X-ray fluorescence analyzer (PANalytical B. V., Almelo, Holland) are listed in Table 1. The designed formulas of the samples are given in Table 2, which contain 100, 95, 90, 85 wt% in-situ synthesized cordierite contents and 0, 5, 10, 15 wt% spodumene, assigned as A1, A2, A3 and A4, respectively. The powder mixtures were dry-mixed for 30 min by the high energy ball mill with the alumina grinding balls. After pretreatments such as pelleting and aging, the mixtures were uniaxially pressed into the cylindrical green body (Φ30 mm × 5 mm) under a pressure of 60 MPa and the rectangular green body (37 mm × 6.50 mm × 6.50 mm) under a pressure of 50 MPa in the stainless steel dies.

Table 2.
Designed formulas of the samples (wt%).

Sample no.	Kaolin	Talc	γ-Al_2O_3	Spodumene	Total
A1	48.86	39.38	11.76	0	100.00
A2	46.42	37.41	11.17	5	100.00
A3	43.98	35.44	10.58	10	100.00
A4	41.53	33.47	10.00	15	100.00

After dried in an oven at 90 °C for 24 h, the samples were pressureless sintered from 1300 °C to 1400 °C for 2 h with an interval of 20 °C in a Si-Mo furnace. In the thermal shock resistance experiment, the samples were heated up from room temperature to 1100 °C with 8 °C/min rate and held for 30 min in a muffle furnace, then the samples were cooled down in air from 1100 °C to room temperature, and returned to heat up to the designed temperature again. When several cycles were completed, the residual bending strength of the samples was measured to evaluate the thermal shock resistance.

The phase compositions of the samples were performed by X-ray diffraction (XRD) using the D/MAX-Ra powder diffractometer (Rigaku, Japan) with Cu Kα (λ=1.54060 Å) radiation in the 2θ range of 5–80° with the scanning speeding of 0.02°/s. The crystalline phases were identified by the powder diffraction patterns of the Inorganic Crystal Structure Database (ICSD). The water absorption (Wa), open porosity (Pa) and bulk density (Db) of the samples were measured by the Archimedes method. The bending strength of the samples was determined by the three points bending method using a computer-controlled electronic universal testing machine (Model RGM-4100, Shenzhen Reger Instrument Co., Shenzhen, China). The thermal expansion coefficients of the samples were obtained by thermal dilatometer (Model WTC-1, Wuhan University of Technology, China). Infrared absorption spectroscopy (IR) was performed by Nexus (Thermo Nicolet, America) spectrophotometer and the resolution was set to 4 cm^{-1} for all the samples. The fracture morphologies of surfaces of the samples were observed by a scanning electron microscope (SEM) (Model JSM-5610LV, Japan). The energy dispersive spectroscopy (EDS) was taken at 15 kV and a vacuum of 9.6×10^{-5} Pa. The fractured surfaces of the samples were etched by 5 wt% hydrofluoric acid for 90 s, and then washed by deionized water and dried at 100 °C to remove glassy phase.

3. Results and discussion

3.1. XRD analysis

The XRD patterns of sample A1 sintered respectively at 1100 °C, 1200 °C and 1300 °C are shown in Fig. 1. The phase analysis of Fig. 1 illustrated that mullite ($3Al_2O_3 \cdot 2SiO_2$, ICSD: 79-1455), protoenstatite ($MgSiO_3$, ICSD: 74-0816), spinel ($MgAl_2O_4$, ICSD: 70-

Table 1.
Chemical compositions of the raw materials (wt%).

Raw materials	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	Li_2O	I.L	Total
Kaolin	48.02	37.84	0.1	0.63	0.07	0	0	0	0	12.96	99.62
Talc	56.51	2.11	0.98	0.09	1.31	30.69	0.0069	0.018	0	7.44	99.155
γ-Al_2O_3	0.35	97.6	0.014	0	0.03	0	0.034	0.33	0	1.49	99.848
Spodumene	68	23.4	0.5	0	0.21	0.2	0.3	0.5	6.2	0	99.41

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