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Phase transformation and properties of high-quality mullite ceramics synthesized using desert drift sands as raw materials

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ABSTRACT

The desert drift sands were used as raw materials to synthesize mullite ceramics in order to save mineral resources and protect the environment. The experimental results show the formation temperature of mullite was reduced significantly and the density and mechanical properties were remarkably improved compared to the reference sample prepared using analytical reagents. The impurity cations formed low thermal expansion of glass phase, contributing to the decrease of thermal expansion coefficient. Finally, the obtained mullite ceramics had excellent densification, mechanical properties and low thermal expansion coefficient.

1. Introduction

In recent years, rapid development of ceramic industry has caused a sharp drop in mineral resources and serious pollution on the environment. Desert drift sands have considerable reserves, but are rarely used in industry due to their low content of quartz (60–80%) [1]. Therefore, it is necessary to find suitable substitutes for mineral materials and to further develop desert resources for the current situation. If using drift sands as raw materials to replace dwindling quartz minerals such as high purity quartz sand and clay, the production costs of mullite ceramics can be drastically reduced, which is beneficial to the green and sustainable development of ceramic industry. In addition, the deserts are used commercially and their surrounding ecological environment will be improved.

Mullite ceramics ($Al_6Si_2O_{13}$) are often used as refractories due to their high melting point (1850 °C), high mechanical properties, low coefficient of thermal expansion (CTE, 5.13×10^{-6}/°C, 25–1000 °C) and good creep resistance [2]. The high quality of mullite ceramics can be obtained, however, only by means of high pure raw materials, advanced sintering process and high sintering temperature (1500–1650 °C) [3–5]. In this paper, the mullite is synthesized from drift sands and commercially available alumina powder, aiming to reduce the sintering temperature and obtain high density, good mechanical and thermal expansion properties.

2. Materials and methods

The drift sands were taken from the desert of Inner Mongolia in China and their chemical compositions (wt%) were 78.91 SiO_2, 9.50 Al_2O_3, 2.33 CaO, 2.26 Fe_2O_3, 2.08 K_2O, 2.00 Na_2O, 0.31 TiO_2, 2.61 Others. Industrial Al_2O_3 (99.7%, Hanjin New Material Co., Ltd., China, Average particle size: 50 μm) was mixed with drift sands (Average particle size: 154 μm) to produce three compositions of mullite ceramics: Mu-1 (Stoichiometric composition, wt%: 34.26 Drift sands, 65.74 Al_2O_3), Mu-2 (Quartz-rich composition 1, wt%: 40.96 Drift sands, 59.04 Al_2O_3) and Mu-3 (Quartz-rich composition 2, wt%: 47.80 Drift sands, 52.20 Al_2O_3). In addition, a reference sample ARM (AR: Analytical Reagent, M: Mullite) with stoichiometric composition was prepared using analytical reagents of SiO_2 (99.0%) and Al_2O_3 (99.9%, XiLong Chemical, China).

The mixed raw materials were milled to an average particle size of 20 μm using water as a medium in a planetary ball mill for 3 h. Then, the powder was pressed into cylinder under an unidirectional pressure of 30 MPa using a pressing machine. Finally, the samples were sintered at 1100–1600 °C for 2–8 h. The ceramics were characterized using RIGAKU D/MAX-2500/PC (XRD, Cu Kα radiation, 40 kV, 100 mA), HORIBA EMAX (EDS), NETZSCH STA 409 PC Luxx (DSC, 1400 °C, 10 K/min), FEI Quanta 650 FEG and JEOL JCM-6000 NeoScope (SEM), NETZSCH/DIL 402PC (thermal

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dilatometer, 30–900 °C, 5 K/min), Jinan Shijin WDW-30 (flexural strength, 40 × 4 × 3 mm, 0.5 mm/min, span of 30 mm), TaiMing, HVS-30Z/LCD (Vickers hardness, 2.5 kgf). The phase contents and lattice parameters were calculated using MDI JADE via a whole pattern fitting method, peak locations and Miller indices, respectively [6]. The density and apparent porosity were tested according to the Archimedes method.

3. Results and discussion

Drift sands consisted of quartz (JCPDS: 85-0797) and feldspar (Anorthite, JCPDS: 41-1481 and others) containing a great number of impurity cations (Fig. 1a). The cell volumes in drift sands were 0.04% (RT, room temperature) and 0.38% (1100 °C) larger than those in pure quartz, respectively, which indicates the quartz lattice is more likely to expand in drift sands (Fig. 1b). Meanwhile, an obvious up-convex baseline (2θ: 20–38°) confirmed the formation of glass phase (Fig. 1a). In addition, the DSC curves (Fig. 1c) show peak T2 and T4 only existed in drift sands. The endothermic peak T1 was related to the transformation from low-quartz to high-quartz. The peak T2 was caused by the transformation of high-quartz into high-tridymite and was slightly below the standard transformation temperature (870 °C). The peaks T3 was related to the transformation of cristobalite. There is evidence that it is almost impossible for the transformation of quartz into tridymite, but quartz can turn directly into cristobalite in the absence of impurity cations. The decrease of transformation temperature is due to a drop in lattice activation energy caused by impurity cations. The endothermic peak T4 can be attributed to the melting of feldspar mixture. Finally, drift sands are mainly composed of quartz and glass phases through sintering at high temperature.

Fig. 2 shows that Mu-1–Mu-3 consisted of α-quartz (JCPDS: 86-1630), feldspar (JCPDS: 70-0287) and corundum (JCPDS: 74-1081) sintered at 1100 °C for 2 h. A new phase of mullite (JCPDS: 82-0037) formed in Mu-1–Mu-3 sintered at 1200 °C for 2 h and the content of mullite reached to the maximum above 1550 °C. In addition, the up-convex baselines showed feldspar formed liquid at 1100 °C. The sand/alumina proportion in Mu-1 facilitated the formation of mullite while an excess of sand with respect to alumina tended to slightly increase glass and corundum and decrease the proportion of mullite (Fig. 2e). The mullite phase did not form in ARM until the temperature was above 1500 °C for 2 h, which showed drift sands as raw materials made the formation temperature of mullite phase fall by 300 °C.

Usually, the liquid phase occurs before the formation of mullite and then two mullitizations appear during the formation of mullite [7]. Drift sands as raw materials contributes to the increase of liquid phase and the decrease of diffusion energy of Al_2O_3 in SiO_2, the formation temperature of liquid phase is reduced greatly and mullitization process is promoted significantly [8,9].

Fig. 3a–d show a small number of closed pores existed in Mu-1–Mu-3 while a large number of open pores were distributed in ARM.

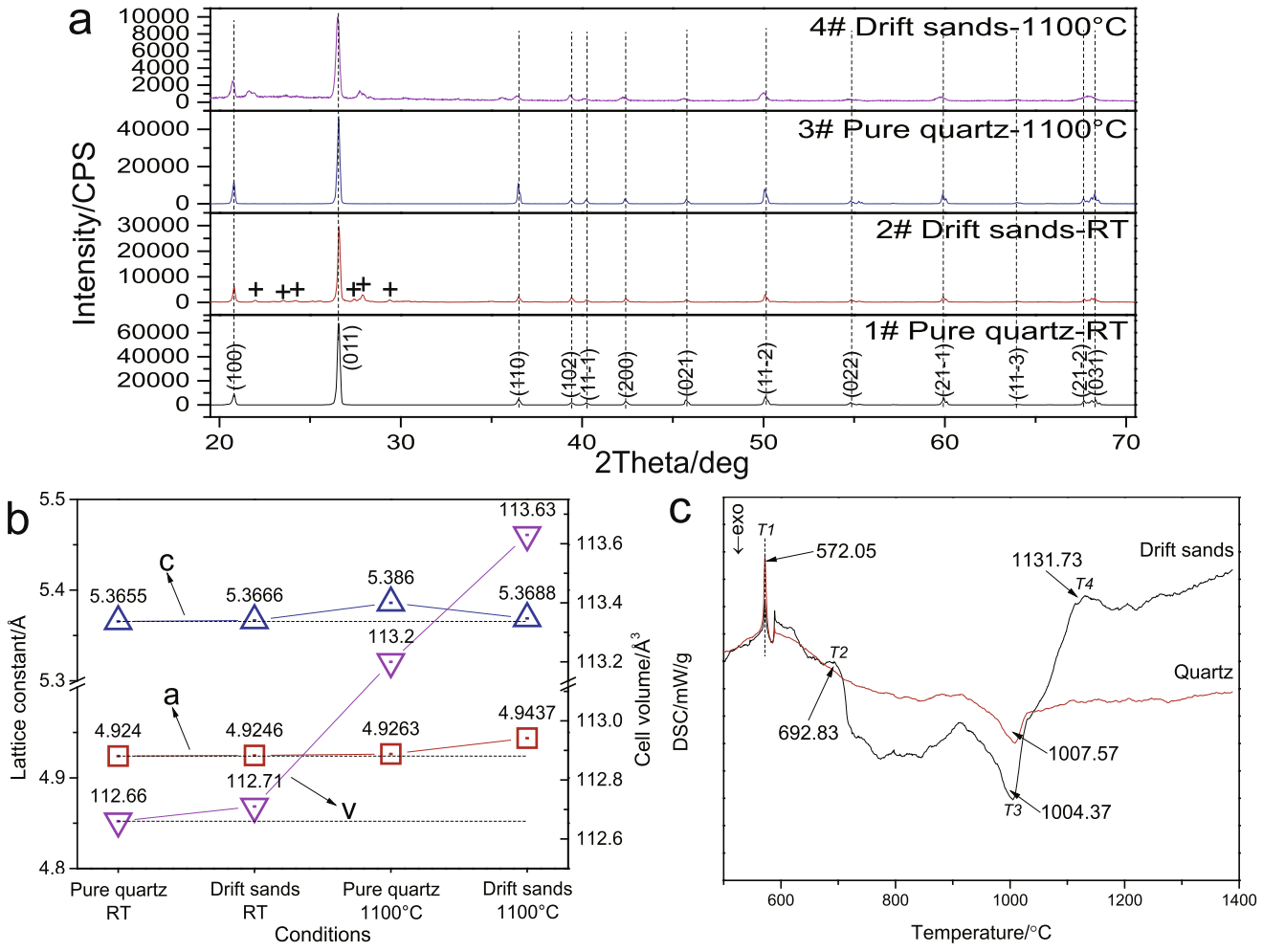

Fig. 1. (a) XRD patterns (unmarked peaks: quartz, +: feldspar), (b) Lattice parameters and (c) DSC curves of drift sands and quartz.