Materials Chemistry and Physics 148 (2014) 449–456

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Influence of particle size on sinterability, crystallisation kinetics and flexural strength of wollastonite glass-ceramics from waste glass and fly ash

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HIGHLIGHTS

- Fine powder exhibited fast sintering rate and high densification/crystallinity.
- The sintering kinetics agrees with the linear shrinkage analysis.
- Fine powder has a smaller Q and larger E_c.
- The mechanical enhancement of glass-ceramics resulted from multiple factors.

ARTICLE INFO

Article history:
Received 25 October 2013
Received in revised form
29 July 2014
Accepted 6 August 2014
Available online 27 August 2014

Keywords:
Glasses
Sintering
Powder diffraction
Crystallisation
Mechanical properties

ABSTRACT

Wollastonite glass-ceramics were prepared from waste glass and fly ash by using the primary powder sintering. The sinterability, crystallisation kinetics and mechanical property of glass-ceramics have been investigated systematically with respect to the influence of particle size of raw materials. As compared to coarse powders (44 μm), fine powders (6 μm) exhibited fast sintering rate, high densification and high degree of crystallinity. The sintering kinetic analysis showed that the sintering activation energies were 229 and 157 kJ mol^{-1} for coarse and fine powders respectively, in well agreement with those from the linear shrinkage analysis. The activation energies of crystallisation were 245 and 317 kJ mol^{-1} for coarse and fine powders, respectively, suggesting high energy barrier of crystallisation for fine powders. The flexural strengths of wollastonite glass-ceramics from fine powders were increased by more than 50% as compared to that from coarse powders, resulting from the improved densification, high degree of crystallinity and crystal shape anisotropy as well as crystal refinement.

1. Introduction

In the past decade, recycling of solid wastes has attracted considerable attention due to the environmental protection and sustainable utilization of natural resources [1,2]. In this respect large amounts of waste glassware and fly ash should be recycled to produce high value-added products such as novel stoneware tiles and functional ceramics [3–5]. The development of sintered glass-ceramic products with waste glass from window glass and glass-ware, as well as fly ash from domiciliary waste incinerators and coal power plants, has become a hot topic of relevant researches.

The variation of particle size exerted significant effects on the densification, phase composition and physical properties of sintered glass-ceramics [6–8]. Fine powders usually imply low sintering temperatures and high densities, and are preferable to prepare functional ceramics with high performance. However, extremely fine powder would obstruct the complete densification of wollastonite glass-ceramics [9]. As for the crystallisation behaviour, iron-rich glass from nickel leaching wastes with an average particle size >100 μm showed three-dimensional crystal growth controlled by diffusion, whereas a particle size <100 μm led to a two-dimensional growth of crystallites [10]. Moreover, fine powders brought about high degrees of crystallinity of glass-ceramics [11]. So far, the sintered glass-ceramics have mostly been prepared from glassy powders [12,13], where the high-temperature melting and subsequent sinter-crystallisation of glassy powders required high energy consumption. In the case of

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http://dx.doi.org/10.1016/j.matchemphys.2014.08.013
0254-0584/

energy saving, direct sintering with primary powders is preferable, especially for the reutilization of waste materials.

In this study, wollastonite glass-ceramics were prepared from waste glass and fly ash with two different particle sizes. The effect of particle size on the sinterability, crystallisation kinetics, phase composition and flexural strength of glass-ceramics was examined in detail. These investigations make for the full comprehension of the fundamental characteristics of sintered glass-ceramics relevant to particle size, shedding light on the industrial application of waste materials.

2. Experimental procedure

2.1. Preparation of powder mixtures

The raw materials included waste glass, fly ash and lime (chemical reagent), the contents of which were determined on the basis of the typical composition of wollastonite glass-ceramics ($72SiO_2$:$8Al_2O_3$:$20SiO_2$). The ordinary soda-lime-silica glass cullet was first melted in a high-purity (>99.99%) alumina crucible at 1450 °C for 2 h, and then quenched into distilled water. The collected glass frits were dried at 120 °C overnight, and pulverized in a planetary mill for 1 h to obtain glass powders. Fly ash powders from thermal power plant were calcined at 800 °C for 2 h to remove any volatiles. Both glass and fly ash powders were separately sieved into two size fractions, i.e., coarse powder (+80–120 mesh) and fine powder (+325 mesh). After that, powder mixtures (coarse/coarse or fine/fine) with 70 wt% waste glass and 30 wt% fly ash were uniformly blended in a mortar with pestle for 30 min, and passed through an 80-mesh screen. The median particle size (d_{50}) of powder mixtures was determined by a laser particle size analyzer (LS230, Coulter) to be 44 μm for the coarse powder mixture and 6 μm for the fine powder mixture, as shown in Fig. 1.

2.2. Preparation of glass-ceramics

The starting materials of glass-ceramics consisted of 15 wt% lime and 85 wt% powder mixture according to the typical composition of wollastonite glass-ceramics. These composite powders were granulated by mixing with a 5 wt% polyvinyl alcohol (PVA) aqueous solution, dried at 120 °C for 1 h, and then uniaxially pressed at 100 MPa to obtain rectangular bars. After drying at 220 °C for 2 h, powder compacts were thermally treated at 550 °C for 2 h to remove the PVA binder, and subsequently followed by sintering at different temperatures in a temperature-programmed muffle furnace with a heating rate of 10 °C min^{-1}. Powder compacts sintered at 950, 1000, 1050 and 1100 °C were labelled as C950, C1000, C1050 and C1100 for coarse powders, and in the same way F950, F1000, F1050 and F1100 for fine powders.

2.3. Characterization

The bulk density of sintered glass-ceramics was measured using the Archimedes' method (ASTM C373-88). The sintering kinetics of powder compacts was evaluated by the variation of bulk density and linear shrinkage during the sintering process. The non-isothermal crystallisation kinetics of powder compacts was quantitatively analyzed by differential scanning calorimetry (DSC, STA-200) using the Kissinger equation. The crystalline structure of sintered glass-ceramics was identified by X-ray diffraction (XRD, Brucker D8 Advance) using Cu K_α radiation, and the degree of crystallinity was estimated from the XRD pattern simulation using the MDI JADE 5.0 software.

The microstructure observation and elemental analysis of glass-ceramics were performed with an environmental scanning electron microscope (SEM/EDS, Quanta 200). The sample surfaces were polished and then chemically etched by immersion in a 2 vol% HF aqueous solution for 20 s. After that, the samples were ultrasonically washed with distilled water and dried in vacuum. The three-point flexural strength of glass-ceramics was measured using rectangular bars (45 mm × 4 mm × 3 mm) in an Instron-type apparatus (WDW-50) with a crosshead speed of 0.5 mm min^{-1}. All rectangular bars were carefully polished to a 6 μm finish and chamfered by using SiC abrasive papers and diamond paste. Each data point represents the average value of at least 5 individual tests.

3. Results and discussion

3.1. Sintering kinetics of glass-ceramics

Fig. 2 shows the relationship of bulk density of glass-ceramics with sintering temperature and soaking time. The sintering activity of fine powders is apparently superior to that of coarse powders under the same conditions. The sintering process of fine powder compacts was nearly complete after 60 min, whereas the sintering process of coarse powder compacts continued even after 120 min. Another phenomenon noticeable is that with the increase in sintering temperature, the bulk density of coarse powder compact varies much more predominantly with soaking time, suggesting

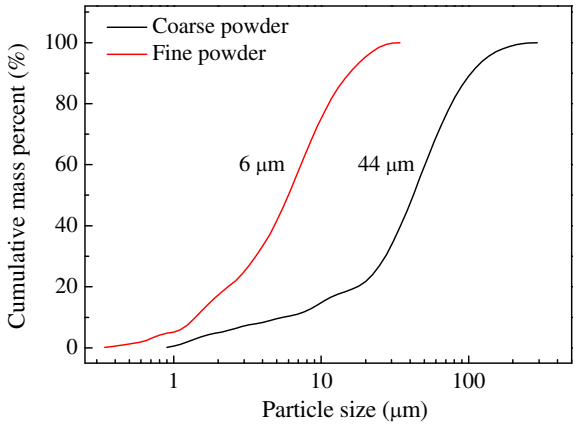

Fig. 1. Particle size distributions of powder mixtures of glass and fly ash.

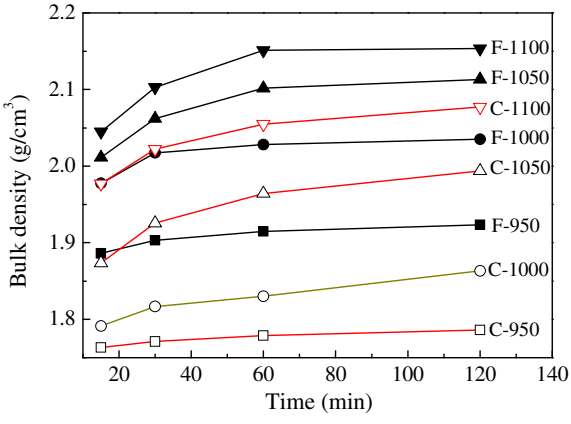

Fig. 2. Bulk densities of glass-ceramics sintered at different temperatures and soaking times.