Glass-ceramics fabricated by efficiently utilizing coal gangue

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ABSTRACT
Glass-ceramics with high strength and lightweight were developed by comprehensively utilizing coal gangue and clay. This work optimized the many processing parameters including the rate of using coal gangue, coal gangue to clay ratio, mineralizers, and process of forming and sintering. Using coal gangue of 75% and coal gangue to clay ratio of 3/1 made the mechanical properties optimal. The sintering temperature of 1370 °C was found to be optimal because mullite and spinel become the main crystal phase and thereby realizing high strength, low water absorbance, and low density for the sintered glass-ceramics. The mechanical properties of the glass-ceramics were further enhanced by adding mineralizer (TiO2, ZnO, and MnO2/dolomite) with reasonable amounts. Moreover, the properties were enhanced by optimizing processing parameters. We developed an excellent approach to sinter glass-ceramics illustrated high strength, lightweight, and low water absorption by efficiently utilizing and managing waste minerals. The sintered glass-ceramics can be used as high-performance proppant materials, cooking ceramics, and building materials.

1. Introduction
Glass-ceramics are widely used as machinable ceramics, cooking ceramics, building materials, electrical ceramics, optical materials, bio-ceramics, glass semiconductors in thermal insulation, etc [1–12]. Some applications require the glass-ceramics to be high strength and lightweight. From a point of the raw material, Comprehensively utilizing the industrial solid wastes with high contents of silica and alumina as raw materials to fabricate the glass-ceramics is attracted because of the advantage in environment management and economics.

Glass-ceramics mainly make up SiO2, Al2O3, CaO, and/or MgO. Many waste minerals contain similar components and so can be potentially used as raw materials for fabricating glass-ceramics. Update, various waste minerals such as incinerator ash, coal ash, slags, waste glass, phosphorus fertilizer, and oil shale ash have been used. Coal gangue as a waste mineral has been widely utilized to fabricate cement [13–15], ceramics [16–18], refractory brick [19], zeolite [20], lightweight aggregate [21], mullite [22], and so on. The coal gangue is extensively produced in coal production. Its amount produced in China is reached above several million tones and increases to about one million tones per year. Currently, the rate of using coal gangue is very limited. Efficiently utilizing the coal gangue is an urgent assignment to reduce air and river pollution and save natural resources. The coal gangue contains high silica of ~62 wt.% and high alumina of ~30 wt.% and thereby is a very suitable raw material to fabricate the high-performance glass-ceramics. The crystal phase and its size and content in the sintered glass-ceramics are critical factors determining the properties. In general, mullite and other crystals composed of both aluminum and silicon will be favorable for the high strength. Meanwhile, the nucleating agent and some process parameters are the deterministic factors for the size and content of the formed crystal phase as well as the density of the sintered glass-ceramics.

This work focuses on the sintering and the property of the glass-ceramics by efficiently utilizing the coal gangue as the main raw material and optimizing processes. The mullite was expected to be the main crystal phase by selecting the reasonable prescriptions of the glass-ceramics in the condition of considering the maximal utilization rate of the coal gangue, The optimization of the nucleating agent and process parameters were focused to further realize the enhancement of property of the sintered glass-ceramics.

2. Experimental methods
Coal gangue as a main raw material was first used. Adding clay aimed to increase the content of Al2O3 and to enhance slurry formability. TiO2, ZnO, Fe2O3, MnO2, BaCO3, MgCO3, and dolomite were used as mineralizers. The sodium tripolyphosphate (STPP) and methyl cellulose (CMC) as a water reducer was utilized to reduce the slurry viscosity. The chemical compositions are listed in Table 1 for all the mineral raw materials.

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Table 1. The composition of the raw materials (wt%).

| Raw material | SiO₂ | Al₂O₃ | CaO | MgO | Fe₂O₃ | TiO₂ | K₂O | Na₂O | Incineration loss |
|--------------|------|-------|-----|-----|-------|------|-----|------|------------------|
| Coal gangue  | 62.50| 20.80 | 2.12| 4.83| 3.14  | 0.83 | 3.12| 3.17 | 0.70             |
| Clay         | 41.03| 37.71 | 2.66| 0.46| 0.14  | 0.14 | 0.68| 0.54 | 14.18            |
| Dolomite     | 3.38 | 1.01  | 31.68| 19.61| 0   | 0    | 0   | 0.20 | 0.37             |

Table 2. The prescriptions of samples (wt%).

| No | Coal gangue | clay | Fe₂O₃ | TiO₂ | ZnO | BaCO₃ | MnO₂ | MgCO₃ | Dolomite |
|----|-------------|------|-------|------|-----|-------|------|-------|----------|
| 1* | 75          | 25   | 0     | 10   | 0   | 0     | 0    | 0     | 6        |
| 2* | 75          | 25   | 0     | 12   | 0   | 0     | 0    | 0     | 0        |
| 3* | 75          | 25   | 0     | 6    | 4   | 0     | 0    | 0     | 0        |
| 4* | 75          | 25   | 0     | 6    | 4   | 4     | 0    | 0     | 0        |
| 5* | 75          | 25   | 0     | 6    | 4   | 4     | 1    | 0     | 0        |
| 6* | 75          | 25   | 0     | 6    | 4   | 4     | 1    | 3     | 0        |
| 7* | 75          | 25   | 0     | 1    | 0   | 0     | 6    | 0     | 0        |
| 8* | 75          | 25   | 0     | 1    | 0   | 0     | 6    | 3     | 0        |
| 9* | 75          | 25   | 0     | 1    | 0   | 0     | 6    | 0     | 6        |
| 10*| 75          | 25   | 0     | 6    | 4   | 0     | 3    | 0     | 0        |
| 11*| 75          | 25   | 5     | 3    | 0   | 0     | 1    | 0     | 0        |
| 12*| 75          | 25   | 3     | 3    | 1   | 1     | 1    | 0     | 0        |
| 13*| 75          | 25   | 3     | 3    | 1   | 1     | 3    | 0     | 0        |
| 14*| 72          | 25   | 0     | 6    | 4   | 0     | 3    | 0     | 0        |
| 15*| 72          | 23   | 0     | 6    | 4   | 0     | 3    | 5     | 0        |
| 16*| 72          | 21   | 0     | 6    | 4   | 0     | 3    | 8     | 0        |
| 17*| 72          | 25   | 0     | 6    | 4   | 0     | 3    | 0     | 0        |
| 18*| 72          | 25   | 0     | 6    | 4   | 0     | 0    | 0     | 8        |

Mullite (Al₆Si₂O₁₃) generally has high strength and so was designed as the main crystal phase. Combine with the high utilization rate of the coal gangue, the prescriptions of glass-ceramics were designed as listed in Table 2.

The raw materials were weighted according to the prescriptions in Table 2 and then homogeneously mixed. The mixtures were wet-milled with a ratio of raw materials: ball: water = 1:2.5: (0.3–0.5) for 15 min, and then sifted with 80 mesh sieve (Aperture: 0.180 mm, ASTM). The fine mixtures were undergone a drying of 50 – 70 °C and 3 – 5 h. Subsequently, ~200 g dried fine mixtures were taken for each sample, into which 5 g STPP and 5 g CMC were added. By further wet-milling for 12 min, the slurry was sifted with 80 mesh sieve (Aperture: 0.180 mm, ASTM), leaving the residue of ~0.1% over the sieve. The fine powders were dried at 50–70 °C for 3 – 5 h, ground and then sifted again with a sieve (80 mesh, aperture: 0.180 mm, ASTM). The fine powders were undergone a granulation by spraying water and stirring. The granulated grains were sifted with a sieve (20 mesh, aperture: 0.850 mm, ASTM). The fine grains were further sprayed with water, constantly stirred, and then processed to shape satisfying the requirement of strength measurement. The formed samples were then pressed at 10 – 35 MPa and dried at 50 – 100 °C for 24 h. The large grains on the sieve were dried at 80 °C for 4 h. The dried billet samples used for strength measurement and the dried large billet grains were finally heated to 1330–1400 °C at a heating rate of 5 – 10 °C/min and sintered for 30 – 90 min.

The crystal phase formed in the sintered glass-ceramics was identified by D/Max-2200PC X-Ray diffractometer (XRD, CuKα1, λ = 0.15406 nm, Rigaku, Japan) at room temperature. The micromorphology was observed by S-570 scanning electron microscopy (SEM, Hitachi, Japan). The flexural strength was determined by a PT-1036PC universal material tester (Baoda, Taiwan). The size of the sample was 3 × 6 × 35–40 mm. The measurement was on the basis of the GB/T-1449-2005 Chinese standard. A three-point bending method was utilized. The sample span was 30 mm. The loading speed was 3 mm/min. The water absorption (Aw) of the grain samples was determined by the following relation:

$$Aw = \frac{W_b - W_d}{W_d}$$

Where the $W_d$ is the weights of the appropriate amount of the grains after dried at 110 °C for 3 h, and $W_b$ is the weights of the grains after soaking in the water for 36 h at room temperature followed by erasing the surface water with a wet towel. The density of the grain samples ($d$) was determined by the following relation:

$$d = \frac{m_0}{m_1 - m_2}$$

Where $m_0$ is the mass of the appropriate amount of the grain samples after undergoing the drying at 105 – 110 °C for 3 h, whereas $m_1$ and $m_2$ are the masses of the grains immersing in the water and after withdrawing from the water, respectively. The withdrawn grains underwent the erosion of the surface water with a wet towel. The weight and mass were determined by HZK-FA300 S Electronic balance (HZ, US).

All the property measurements were repeated three times to obtain accurate results.
3. Results and discussion

To select a reasonable rate of utilizing the coal gangue, nine samples with different coal gangue to clay ratio from 50/50 to 90/10 were first tested. Results showed a flexural strength (55–78 MPa) and water absorption (∼5–12%). The optimal ratio of coal gangue to clay is 75/25. Therefore, this ratio was further studied by adding various mineralizers. Table 2 lists the prescriptions. Table 3 summarized the various properties of the glass-ceramics that were formed at the press of 30 MPa and sintered at different temperatures for 30 min. TiO$_2$, ZnO- and MnO$_2$-codoping (prescription 10$^a$) as mineralizers lead to better effects, achieving a maximal flexural strength (180.67 MPa) and minimal water absorption (∼0%). Furthermore, TiO$_2$, ZnO-, and dolomite-codoping (prescription 18$^a$) as mineralizers lead to an optimal effect, achieving a maximal flexural strength (187.67 MPa) and a minimal water absorption (∼0%). In general, higher sintering temperature promotes full solid solution reaction, full crystallization, and impact microstructure. However, the too high sintering temperature could lead to the formation of an excess glass phase. Thus, the optimal sintering temperature is 1370 °C as shown in Table 3. The chemical compositions of the prescription 10$^a$ and 18$^a$ are listed in Table 4.

According to thermodynamics analysis [23], the selected mineralizers obey the nucleation ability sequence of Fe$_2$O$_3$> TiO$_2$> ZnO>MnO$_2$ and the optimal nucleation temperature order of Fe$_2$O$_3$< TiO$_2$< ZnO<MnO$_2$ in a melting amorphous system. This is because the Fe$_2$O$_3$ is of the largest melting entropy that results in its largest decrease in free enthalpy in the ordering process of the melting amorphous system and thereby resulting in maximal nucleation ability and lowest nucleation temperature [23]. The mineralizers could be reacted with other raw materials in the process of the heating for the sintering. During this solid solution reaction and further heating of the formed solid solution, the mineralizer Fe$_2$O$_3$, TiO$_2$, ZnO, and MnO$_2$ acted as nucleating agents, resulting in the formation of extensive smaller nucleus. On the surface of these smaller nuclei, smaller crystalline grains epitaxially grow in the process of the sintering, which makes the samples more and more compact and strong. Enhancement in mechanical properties resulted from crystallization also reported by Mirza et al. [9]. The Fe$_2$O$_3$ failed to achieve better effect because of its too low nucleation temperature. Whereas the TiO$_2$, ZnO, and MnO$_2$ may have suitable nucleation temperatures in the studied systems, thereby leading to the optimal properties of the glass-ceramics.

Figure 1 illustrates the XRD patterns of the glass-ceramics fabricated with prescription 10$^a$ and 18$^a$. The mullite (Al$_6$Si$_2$O$_{13}$) (JCPDS: 15–0776) and spinel ((Zn, Mg)Al$_2$O$_4$) (JCPDS: 73–1961, 73–1959, 77–1193) was identified to be the main crystal phases. The dolomite doping leads to an increase in mullite content and the decrease in a spinel. The mullite is generally formed at a higher temperature, such as above 1320 °C as reported in previous literature [24]. The CaO and MgO in the dolomite may accelerate solid solution reaction among the raw materials and so is favorable for the formation of mullite. The mullite can show a compressive strength (0.69 GPa) that is larger in comparison with many crystals, including the cordierite (0.32 GPa), forsterite (0.55 GPa), and boron nitride (0.32 GPa) [25], thereby resulting in the high strength of the sintered glass-ceramics. Therefore, the glass-ceramics fabricated with prescription 10 and 18$^a$ show optimal properties. Furthermore, the XRD peaks of the mullite phase are enhanced with increasing sintering temperature from 1330 °C to 1370 °C, and so the glass-ceramics sintered at 1370 °C shows the optimal properties.

Table 3. Water absorbance and flexural intensity of glass-ceramics fabricated from 1-20% at various sintering temperatures.

| No. | Water absorbance (%) | Flexural intensity (MPa) |
|-----|---------------------|-------------------------|
|     | 1330°C | 1350°C | 1370°C | 1400°C | 1330°C | 1350°C | 1370°C | 1400°C |
| 1$^a$ | 0 | 0 | 1.87 | 0 | 67.67 | 66.5 | 86.6 | 107.8 |
| 2$^a$ | 1.72 | 0 | 1.54 | 0 | 68.47 | 97.62 | 99.6 | 114.5 |
| 3$^a$ | 0 | 0 | 1.23 | 1.11 | 66.75 | 39.78 | 89.96 | 68.59 |
| 4$^a$ | 1.64 | 1.75 | 0 | 0 | 58.43 | 82.53 | 85.66 | 80.65 |
| 5$^a$ | 1.82 | 1.57 | 0 | 0 | 68.9 | 99.10 | 99.49 | 93.99 |
| 6$^a$ | 0 | 1.87 | 0 | 1.24 | 69.8 | 95.82 | 90.49 | 47.89 |
| 7$^a$ | 0 | 0 | 2.24 | 0 | 98.25 | 89.78 | 100.67 | 90.97 |
| 8$^a$ | 1.56 | 0 | 2.23 | 0 | 49.35 | 99.98 | 99.15 | 86.79 |
| 9$^a$ | 1.26 | 1.26 | 0 | 2.45 | 48.97 | 96.78 | 86.59 | 98.35 |
| 10$^a$ | 0 | 0 | 0 | 0 | 98.66 | 130.09 | 180.67 | 122.65 |
| 11$^a$ | 1.67 | 0 | 2.03 | 0 | 59.8 | 69.8 | 99.8 | 89.8 |
| 12$^a$ | 1.46 | 1.78 | 0 | 0 | 69.8 | 59.87 | 95.56 | 56.78 |
| 13$^a$ | 1.45 | 1.77 | 0 | 0 | 75.66 | 77.89 | 90.99 | 86.8 |
| 15$^a$ | 5.45 | 2.13 | 3.77 | 0 | 100.06 | 107.47 | 92.94 | 68.27 |
| 16$^a$ | 0 | 1.87 | 0 | 1.24 | 108.49 | 105.82 | 127.8 | 95.29 |
| 17$^a$ | 0 | 1.54 | 0 | 0 | 97.62 | 99.49 | 110.99 | 80.03 |
| 18$^a$ | 0 | 0 | 0 | 1.25 | 98.32 | 132.04 | 187.67 | 135.97 |

Table 4. Chemical composition of the smaples (wt%).

|   | SiO$_2$ | Al$_2$O$_3$ | CaO | MgO | TiO$_2$ | Fe$_2$O$_3$ | ZnO | MnO$_2$ | K$_2$O | Na$_2$O |
|---|--------|-----------|-----|-----|--------|-----------|-----|--------|------|-------|
| 10$^a$ | 51.26 | 23.35 | 3.03 | 3.36 | 5.93 | 2.14 | 5.95 | 2.76 | 2.25 | 3.31 |
| 18$^a$ | 51.36 | 22.51 | 4.28 | 4.60 | 3.58 | 2.14 | 5.93 | 0 | 2.26 | 3.34 |
The SEM micrograph of the glass-ceramics fabricated from prescription 10 and 18 at a sintering temperature of 1370 °C is shown in Figure 2. Some rodlike particles can be observed. The rodlike particles are tinier in the glass-ceramics fabricated from prescription 18 than that from prescription 10. These rodlike particles could be the mullite phase. Other granular particles could be the spinel phase and quasi-crystalline solid solution and/or amorphous phase. Some porous can also be observed in Figure 2 (b). This may be ascribed to the volatilization of gas formed from dolomite decomposition. Moreover, the glass-ceramics show impact and connected microstructure, which ensure excellent properties of the glass-ceramics.

To understand the desorption process of the adsorbed water, the thermogravity curve of the glass-ceramics fabricated with prescription 10 was measured and showed in Figure 3. Before the measurement, the glass-ceramics were soaked in the water for 36 h at room temperature and then erased with a wet towel to remove the excess surface water. The heating rate used for the measurement was 10 °C/min. The adsorbed water began to desorbed at ~50 °C, rapidly desorbed from ~90 to ~120 °C, and reached a balance at ~150 °C. Further desorption beginning at ~340 °C could be ascribed to the desorption of chemically adsorbed water and surface hydroxyl.

To optimize the processing parameters, some prescriptions were further studied in the condition of various processing parameters including the granularity of raw materials, ball-milling and drying parameters of the mixtures, the parameters of forming and sintering. The results are shown in Figures 4–8. The decreasing the grain size of the raw materials from 10 mesh to 46 mesh results in greatly enhanced properties of the glass-ceramics (Figure 4). Smaller grain size favors faster solid solution reaction, faster gas volatilization, smaller nuclei...
leading to higher crystallinity, more impact microstructure, and so higher properties. The increase of forming pressure from 10 to 30 MPa results in a great enhancement of the strength. However, further increase of the forming pressure only shows a slight effect (Figure 5). Higher forming pressure leads to more impact contact between the grains and thereby favoring solid solution reaction, and nucleation and crystallization. Besides, higher forming pressure leads to more impact microstructure of the glass-ceramics. The water content could affect the properties after sintering because high water content could result in rapid water volatilization that could result in the deformation and crack of the samples during the heating process. Also, the high water content could produce pores during heating and thereby affect the density and properties. However, experimental results showed that optimal drying temperature and time of drying the billets are found to be 70–80 °C and 5–6 h (Figures 6 and 7), respectively. The increasing sintering time from 30 min to 90 min results in the decrease of density and the slight decrease of flexural strength, so that reasonable sintering time is 30 min (Figure 8). Too long sintering time could result in excess solid solution reaction and glass phase formation, and thereby deteriorating the properties.

To today, the glass-ceramics fabricated by utilizing various wastes [26–30] only showed the bending strength of 30 – 130 MPa. The factors determining the strength mainly involve the type, content, and size of the crystal phases formed in the glass-ceramics. These glass-ceramics showed the density in the range of ~1.8 – 3.0 g/cm³ [26,27,29–32]. The glass-ceramics fabricated with coal gangue contained the gehlenite as a main crystal phase and nepheline as the next crystal phase [16]. The glass-ceramics only showed a bending strength of 28 MPa [16]. The Mullite glass-ceramics fabricated with
coal gangue and γ-Al₂O₃ showed the bending strength from 64 MPa to 218 MPa as adding the La₂O₃ from 0 mol% to 10 mol% [18]. The porous mullite ceramic fabricated from coal gangue and bauxite showed the flexural strength of 66.06 to 133.61 MPa varied with corn starch content [22]. Moreover, previous literature has reported that high crystallinity and low porosity can result in high chemical stability and acid-resistance [9,33].

Figure 6. Variations of flexural strength, water absorbance and density of the glass-ceramics with the temperature of drying the samples. (Prescription: 17º, forming pressure: 30 MPa, sintering temperature: 1370 °C, sintering time: 30 min).

Figure 7. Variations of (a) flexural strength and (b) water absorbance of the glass-ceramics with the time of drying the samples. (Prescription: 17º, forming pressure: 30 Mpa, drying temperature: 70–80 °C, sintering temperature: 1370 °C, sintering time: 30 min).

Figure 8. Variations of flexural strength, water absorbance and density of the glass-ceramics with sintering time. (Prescription: 18º, forming pressure: 30 MPa, drying temperature: 70–80 °C, drying time: 5.5 h, sintering temperature of 1370 °C).
4. Conclusion

The industrial solid waste coal gangue has been efficiently utilized to fabricate the glass-ceramics with high properties. The rate of utilizing the coal gangue, mineral additives, mineralizers, processing parameters of forming and sintering were optimized. The optimal utilization rate of the coal gangue is determined to be 75 wt.% when an appropriate amount of clay (25 wt.%) was used. The reasonable design of the prescription and the optimization usage of the mineralizers of TiO₂, ZnO, and MnO₂/dolomite result in full nucleation and epitaxial growth of rodlike Mullite and spinel crystal phases and so an efficient enhanced property of the glass-ceramics. The co-doping of TiO₂, ZnO, and dolomite leads to the strength as high as ~187.67 MPa, a low density of 1.83 g/cm³, and low water absorption of ~0%. Processing parameter optimization further realizes higher crystallinity, more impact microstructure, higher properties, and lower energy consumption. The optimal forming processing parameters are smaller raw material granularity (46 mesh), the temperature (70 – 82 °C) and time (5 – 6 h) of drying the wet-milled mixtures, and the forming pressure of 30 MPa. Meanwhile, reasonable temperature (1370 °C) and time (30 min) of sintering are found. This work suggested a new approach for the fabrication of the high-performance glass-ceramics by utilizing the industrial solid waste, the fabricated glass-ceramics may be promising lightweight materials for some applications such as proppant materials, cooking ceramics, and building materials, et al.

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