High Strength Glass-Ceramics Sintered With Coal Gangue as a Raw Material

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Abstract: High strength lightweight glass-ceramics were fabricated with coal gangue and clay as main raw materials. The utilization ratio of coal gangue, the ratio of the coal gangue with clay, mineralization agents, forming process and sintering process on the properties of the fabricated glass-ceramics were optimized. The utilization ratio of coal gangue reached 75, and the ratio of coal gangue to clay was 3/1, as an optimal property was observed. The optimal sintering temperature was found to be 1370 °C. At this optimal temperature, the sintered glass-ceramics showed the main phase of mullite and spindle and so showed high strength, low density, and low water absorbance. The appropriate amounts of codoping of the TiO2, ZnO, and MnO2/dolomite as mineralization agents obviously enhanced the properties of the glass-ceramics. Process optimizations further determined reasonable and optimal process parameters. The high strength lightweight glass-ceramics fabricated in this work may be very suitable for various applications including building materials, cooking ceramics, and proppant materials, et al.

Keywords: Coal gangue; Glass-ceramics; Forming process; Sintering process; Property.

1. Introduction

Glass-ceramics are attractive materials used in various applications such as building materials, cooking ceramics, machinable ceramics, bio-ceramics, electrical ceramics, glass semiconductors in thermal insulation, optical materials, etc. [1-10]. High strength and lightweight are required for some applications. From a point of the raw material, utilizing the industrial solid wastes with high contents of silica and alumina is the main direction of developing the glass-ceramics because of low cost and high product performance.

Because the main constituents of glass-ceramics are SiO2, Al2O3, CaO, and/or MgO, many inorganic wastes are potential starting materials. Various wastes have been used, such as slags, coal ash, incinerator ash, waste glass, and shell, oil shale ash and phosph-orous fertilizer. Coal gangue as a type of industrial solid waste is widely utilized to produce cement [11-13], ceramics [14-16], refractory brick [17], zeolite [18], lightweight aggregate [19], mullite [20], and so on. A very large amount of the coal gangue is produced by coal production. The amount produced in China is reached above several million tones and increases to about one million tons per year. The comprehensive utilization rate of the coal gangue currently is now limited. To prevent air and river from pollution and save the resource, the efficient management and full utilization of the coal gangue is urgent. The coal
gangue has a high alumina content of ~30 wt.% and a high silica content of ~62 wt.%, and so is suitable for fabrication of the high strength glass-ceramics.

For sintering high strength glass-ceramics, the type, size, and content of crystal phase formed in the sintered samples are critical factors. In general, mullite and other crystals composed of both aluminium and silicon will be favorable for the high strength of the glass-ceramics. 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.

Thus, this work focuses on the fabrication and the property of the glass-ceramics with the coal gangue as a main raw material. The mullite was expected to be a 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 nucleating agent and process parameter were focused to further realize the enhancement of property of the sintered glass-ceramics.

2. Materials and Experimental Procedures

The coal gangue was used as main raw materials. Clay was used to increase the Al₂O₃ content of the glass-ceramics and as an adhesive to improve formability of slurry. TiO₂, ZnO, Fe₂O₃, MnO₂, BaCO₃, MgCO₃, and dolomite were used as mineralizers. The sodium tripolyphosphate (STPP) and methyl cellulose (CMC) was used as a water reducer to decrease the viscosity of the slurry. The chemical composition of the raw materials is listed in Table I.

| Raw material | SiO₂ | Al₂O₃ | CaO | MgO | Fe₂O₃ | TiO₂ | K₂O | Na₂O | Ignition 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    | 0.20| 0.37 | 42.85        |

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

The typical process for fabrication of the glass-ceramics is listed below: First, the raw materials were mixed according to the designed prescriptions. The mixtures were filtered through 10 mesh and 45 mesh (Aperture: 20.5 mm and 0.35 mm, ASTM), respectively, wet-milled at a ratio of raw materials: ball: water = 1: 2.5: (0.3-0.5) for 15 min, and then filtered through 80 mesh (Aperture: 0.180 mm, ASTM). The obtained fine powders were prilled by spraying water and stirring. The prilled grains were filtered through 20 mesh (Aperture: 0.850 mm, ASTM). The fine grains under the sieve were re-sprayed with water with constant stirring and then processed to shape required for 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 samples and grains were finally heated at a heating rate of 5-10 °C/min to 1330-1400 °C and sintered for 30-90 min.
Tab. II The prescriptions of samples (wt%).

| No. | Coal gangue | clay | Fe_2O_3 | TiO_2 | ZnO | BaCO_3 | MnO_2 | MgCO_3 | 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   | 0      | 4     | 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   | 5       | 3     | 1   | 1      | 1     | 0      | 0        |
| 13# | 75          | 25   | 5       | 3     | 1   | 1      | 3     | 0      | 0        |
| 14# | 72          | 25   | 0       | 6     | 4   | 0      | 3     | 3      | 0        |
| 15# | 72          | 25   | 0       | 6     | 4   | 0      | 3     | 5      | 0        |
| 16# | 72          | 25   | 0       | 6     | 4   | 0      | 3     | 8      | 0        |
| 17# | 72          | 25   | 0       | 6     | 4   | 0      | 3     | 0      | 5        |
| 18# | 72          | 25   | 0       | 6     | 4   | 0      | 0     | 0      | 8        |

The structural phase of the glass-ceramics was identified at room temperature using X-Ray diffractometer (XRD, CuKα1, λ=0.15406 nm, Model No. D/Max-2200PC, Japan). The morphology of the glass-ceramics was analyzed by scanning electron microscopy (SEM, Model No: S-570, Japan). The flexural strength of the glass-ceramics was measured by a strength tester (Model No: 401/3, Germany). The water absorption (A_w) of the grain glass-ceramics was calculated by the following equation:

\[ A_w = \frac{W_h - W_d}{W_d} \]

Where the \( W_d \) is the weight of the appropriate amount of the grains after dried at 110 °C for 3 h, the \( W_h \) is the weight of the above-dried grains after soaked in water at room temperature for 36 h. The density of the grain glass-ceramics (d) was calculated by the equation:

\[ d = \frac{m_0}{m_1 - m_2} \]

Where \( m_0 \) is mass of the appropriate amount of the grains after drying at 105-110 °C for 3 h, \( m_1 \) and \( m_2 \) are masses of the grains after immersed in water and the grains immersing in water at room temperature, respectively.

3. Results and Discussion

To select a reasonable proportion of the coal gangue, five samples with a different ratio of coal gangue with clay were first tested. Results indicated that the optimal ratio of coal gangue with clay is 75/25. Therefore, this ratio was further studied by adding various mineralizers. Table II lists the prescriptions. Table III summarized the properties of the glass-ceramics formed at the press of 30 MPa and sintered at different temperatures for 30 min. TiO_2-, ZnO- and MnO_2-codoping (prescription 10#) lead to better effects. A minimal water absorption (~0 %) and maximal flexural strength (180.67 MPa) are achieved. Furthermore, TiO_2-, ZnO-, and dolomite-codoping (prescription 18#) lead to an optimal effect. A minimal
water absorption (~0 %) and maximal flexural strength (187.67 MPa) are achieved. 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 a glass phase. Thus, the optimal sintering temperature is 1370 °C. The chemical compositions of the prescription 10# and 18# are listed in Table IV.

**Tab. III** 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 | 1330 °C | 1350 °C | 1370 °C | 1400 °C |
| 1°  | 0       | 0       | 1.87    | 0       | 67.67   | 66.5    | 86.6    | 107.8   | 0       | 0       | 2.24    | 0       | 98.25   | 98.79   | 100.67  | 90.97   |
| 2°  | 1.72    | 0       | 1.54    | 0       | 68.47   | 97.62   | 99.6    | 114.5   | 0       | 0       | 2.23    | 0       | 49.35   | 99.98   | 99.15   | 86.79   |
| 3°  | 0       | 0       | 1.23    | 1.11    | 66.75   | 39.78   | 89.96   | 68.59   | 0       | 1.24    | 69.8    | 95.82   | 90.49   | 47.89   | 98.35   |
| 4°  | 1.64    | 1.75    | 0       | 0       | 58.43   | 82.53   | 85.66   | 80.65   | 0       | 0       | 1.24    | 69.8    | 95.82   | 90.49   | 47.89   |
| 5°  | 1.82    | 1.57    | 0       | 0       | 68.9    | 99.10   | 99.49   | 93.99   | 0       | 0       | 1.24    | 69.8    | 95.82   | 90.49   | 47.89   |
| 6°  | 0       | 1.87    | 0       | 0       | 2.24    | 0       | 98.25   | 98.79   | 100.67  | 0       | 2.45    | 48.97   | 96.78   | 86.59   | 98.35   |
| 7°  | 0       | 0       | 2.24    | 0       | 98.25   | 98.79   | 100.67  | 90.97   | 0       | 0       | 2.45    | 48.97   | 96.78   | 86.59   | 98.35   |
| 8°  | 1.56    | 0       | 2.23    | 0       | 49.35   | 99.98   | 99.15   | 86.79   | 0       | 0       | 2.45    | 48.97   | 96.78   | 86.59   | 98.35   |
| 9°  | 1.26    | 1.26    | 0       | 0       | 2.45    | 48.97   | 96.78   | 86.59   | 0       | 0       | 2.45    | 48.97   | 96.78   | 86.59   | 98.35   |
| 10° | 0       | 0       | 0       | 0       | 98.66   | 130.09  | 180.67  | 122.65  | 0       | 0       | 0       | 98.66   | 130.09  | 180.67  | 122.65  |
| 11° | 1.67    | 0       | 2.03    | 0       | 59.8    | 69.8    | 99.8    | 89.8    | 0       | 0       | 0       | 98.66   | 130.09  | 180.67  | 122.65  |
| 12° | 1.46    | 1.78    | 0       | 0       | 69.8    | 59.87   | 95.56   | 56.78   | 0       | 0       | 0       | 98.66   | 130.09  | 180.67  | 122.65  |
| 13° | 1.45    | 1.77    | 0       | 0       | 75.66   | 77.89   | 90.99   | 86.68   | 0       | 0       | 0       | 98.66   | 130.09  | 180.67  | 122.65  |
| 14° | 5.45    | 2.13    | 3.77    | 0       | 100.06  | 107.47  | 92.94   | 68.27   | 0       | 0       | 0       | 98.66   | 130.09  | 180.67  | 122.65  |
| 15° | 1.75    | 0       | 0       | 1.85    | 102.65  | 101.66  | 80.65   | 78.95   | 0       | 1.24    | 108.49  | 105.82  | 127.8   | 95.29   | 80.03   |
| 16° | 0       | 1.87    | 0       | 1.24    | 108.49  | 105.82  | 127.8   | 95.29   | 0       | 0       | 1.25    | 97.62   | 99.49   | 110.99  | 80.03   |
| 17° | 0       | 1.54    | 0       | 0       | 97.62   | 99.49   | 110.99  | 80.03   | 0       | 0       | 1.25    | 98.32   | 132.04  | 187.67  | 135.97  |
| 18° | 0       | 0       | 0       | 1.25    | 98.32   | 132.04  | 187.67  | 135.97  | 0       | 0       | 1.25    | 98.32   | 132.04  | 187.67  | 135.97  |

**Tab. IV** Chemical composition of the samples (wt%).

|      | SiO₂  | Al₂O₃ | CaO  | MgO  | Fe₂O₃ | TiO₂  | ZnO  | MnO₂ | K₂O  | Na₂O  |
|------|-------|-------|------|------|-------|-------|------|------|------|-------|
| 10°  | 51.26 | 23.35 | 2.03 | 3.36 | 2.14  | 5.95  | 3.59 | 2.76 | 2.26 | 3.31  |
| 18°  | 51.36 | 22.51 | 4.28 | 4.60 | 2.14  | 5.93  | 3.58 | 0    | 2.26 | 3.34  |

Based on thermodynamics [21], the nucleating agents selected in this work follows a nucleation ability order of Fe₂O₃>TiO₂>ZnO>MnO₂ and an optimal nucleation temperature order of Fe₂O₃<TiO₂<ZnO<MnO₂.

The solid solution reaction could take place among the nucleating agents and some components, such as SiO₂, Al₂O₃, CaO, MgO, etc, in the process of the heating for the sintering. During this reaction and further heating of the formed solid solution, the Fe₂O₃, TiO₂, ZnO, and MnO₂ could result in the formation of a large amount of smaller nucleus. On these smaller nuclei, a large amount of smaller crystalline epitaxially grow, which leads to more compact microstructure and higher strength. Enhancement in mechanical properties resulted from crystallization also reported by Mirza et al [22]. TiO₂, ZnO, and MnO₂ may have suitable nucleation temperature in the studied systems, thereby leading to optimal properties of the glass-ceramics.
Fig. 1. XRD patterns of the glass-ceramics fabricated at different sintering temperatures for 30 min (Prescription: 10# and 18#, forming pressure: 35 MPa).

Fig. 1 shows the XRD patterns of the glass-ceramics fabricated with prescription 10# and 18#. The main crystal phase in the glass-ceramics is 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). The dolomite doping leads to increase in mullite content and decrease in a spindle. The mullite is generally formed at a higher temperature, such as above 1320 °C as reported in previous literature [23]. 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 has a larger compressive strength (0.69 GPa) than many crystals, such as the forsterite (0.55 GPa), the cordierite (0.32 GPa) and boron nitrate (0.32 GPa) [24], and so can provide high strength for the glass-ceramics. Thus, the glass-ceramics fabricated with prescription 10 and 18# show optimal properties. Furthermore, the XRD peaks of the mullite phase are enhanced with increasing sintering temperature from 1330 to 1370 °C, and so the glass-ceramics sintered at 1370 °C shows the optimal properties.

The SEM micrograph of the glass-ceramics fabricated from prescription 10# and 18# at a sintering temperature of 1370 °C is shown in Fig. 2. Some rodlike particles can be observed. The rodlike particles are tinier in the glass-ceramics fabricated from prescription
than that from prescription 10#. These rodlike particles could be 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 Fig. 2b. 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.

Fig. 2. SEM micrographs of the glass-ceramics fabricated at the sintering temperature of 1370 °C for 30 min (Prescription: 10# and 18#, forming pressure: 35 MPa).

Fig. 3. Flexural strength and water absorbance of the glass-ceramics fabricated with the raw materials with the different particle size of raw materials. (Prescription: 15#, forming pressure: 30 MPa, sintering temperature: 1370 °C, sintering time: 30 min).

To optimize processing parameter, some prescriptions were further studied in the condition of various processing parameters including grain size of raw materials, drying temperature and drying time of the milled powders, forming pressure, and sintering time. The results are shown in Fig. 3-7. The decreasing the grain size of the raw materials from 10 mesh to 45 mesh results in greatly enhanced properties of the glass-ceramics (Fig. 3). 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 optimal temperature and time of drying the wet-milled powders are 70-80 °C and 5-6 h (Fig. 4 and
Similarly, the increase of forming pressure from 10 to 30 MPa results in a great enhancement of the properties of the glass-ceramics.

![Graph](image)

**Fig. 4.** Variations of flexural strength, water absorbance and density of the glass-ceramics with the temperature of drying wet-milled powders. (Prescription: 17°, forming pressure: 30 MPa, sintering temperature: 1370 °C, sintering time: 30 min).

However, further increase in forming pressure only shows a slight effect (Fig. 6). Higher forming pressure leads to more impact contact between the grains and thereby favoring solid solution reaction, and nucleation and crystallization. In addition, higher forming pressure leads to more impact microstructure of the glass-ceramics. The increasing sintering time from 30 to 90 min results in the decrease of density and the slight decrease of flexural strength, so that reasonable sintering time is 30 min (Fig. 7). Too long sintering time could result in excess solid solution reaction and glass phase formation, and thereby deteriorating the properties.

![Graph](image)

**Fig. 5.** Variations of (a) flexural strength and (b) water absorbance of the glass-ceramics with the time of drying wet-milled powders. (Prescription: 17°, forming pressure: 30 MPa, drying temperature: 70-80 °C)
Fig. 6. Variations of flexural strength, water absorbance and density of the glass-ceramics with forming pressure. (Prescription: 17#, forming pressure: 30 MPa, drying temperature: 70-80 °C, drying time: 5.5 h, sintering temperature of 1370 °C, sintering time: 30 min).

Fig. 7. Variations of flexural strength, water absorbance and density of the glass-ceramics with sintering time. (Prescription: 18#, forming pressure: 30 MPa, drying temperature: 70-80 °C, drying time: 5.5 h, sintering temperature of 1370 °C).

To today, the glass-ceramics prepared with various wastes by many researchers [25-29] generally showed a bending strength range of 30-130 MPa. The type, content, size, and density of the crystal phases are the determining factors. The density of these glass-ceramics was in the range of ~1.8-3.0 g/cm³ [25, 26, 28-31]. Moreover, the glass-ceramics prepared in this work contain relatively small amounts of alkali metal and alkali earth metal cations (i.e. Na₂O, K₂O, CaO, and Mg). This can provide an excellent water-resistance and acid-resistance. Relative high Al₂O₃ content can provide a good alkali-resistance. The mullite crystal phase and low porosity (low water absorption) are also in the favor of these chemical stabilities. Previous literature has reported that high crystallinity and low porosity can result in high chemical stability and acid-resistance [22, 32].
4. Conclusion

The coal gangue as an industrial solid waste has been efficiently used to fabricate the glass-ceramics with high properties. The utilization rate of 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 mineralizers of TiO₂, ZnO, and MnO₂/dolomite codoping result in full nucleation and epitaxial growth of rodlike mullite and spindle crystal phases and so an efficient enhanced property of the glass-ceramics. The co-doping of TiO₂, ZnO, and dolomite leads to an optimal strength of ~187.67 MPa, a water absorption of ~0 %, and a density of 1.83 g/cm³. Processing parameter optimization further realizes higher crystallinity, more impact microstructure, higher properties, and lower energy consumption. The optimal forming processing parameters are smaller particle size of raw materials (45 mesh), the temperature and time of drying the wet-milled powder (70-82 °C, 5-6 h), and the forming pressure of 30 MPa. Meanwhile, reasonable sintering temperature and sintering time are 1370 °C and 30 min, respectively. The glass-ceramics fabricated with the raw materials and the processes present in this work may be promising lightweight materials for various applications including building materials, cooking ceramics, and proppant materials, et al.

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