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# Effect of binder on the structure and mechanical properties of lightweight bubble alumina ceramic

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**Abstract**

The effect of binders such as ammonium aluminum sulphate, phosphoric acid and composite binder on the properties of lightweight bubble alumina ceramic was studied. The composite binder was composed of ammonium aluminum sulphate and phosphoric acid. Ammonium aluminum sulphate solution can improve compressive strength of alumina bubbles effectively but can not improve that of lightweight bubble alumina ceramic due to the fewer nano-alumina powders in situ decomposed of ammonium alumina sulphate. Trans-ball fractures occurred in thermal shock test. Phosphoric acid solution can improve compressive strength of alumina bubble ceramic because of promoting sintering properties of aluminum phosphate in situ produced by phosphoric acid and alumina component during sintering but decrease that of alumina bubbles. Along-ball fractures occurred in thermal shock test. The composite binder combined with the advantages of ammonium alumina sulphate and phosphoric acid and improved the compressive strength of both alumina bubbles and lightweight bubble alumina ceramic, and effectively reduce the amount of the binders and lower the product cost. At the sintering temperature of 1700 °C, with composite ammonium alumina sulphate and phosphoric acid as binder, the density of lightweight bubble alumina ceramic was between 1.20 and 1.60 g/cm$^3$, and the compressive strength was 18–42 MPa.


*Keywords:* Alumina bubble; Binder; Ceramic

Bubble alumina ceramic is a high-temperature insulation material with properties of lightweight, high strength and high temperature resistance [1–4]. Compared with the dense refractory materials [4–6], bubble alumina ceramics have many high performances such as light-weight, low density, high thermal shock loading, well-heat insulating and low thermal capacity. While compared with the other common heat-insulating refractory materials [7,8], the bubble alumina ceramics have higher compressive resistance, higher softening temperature under load and lower permanent linear shrinkage. It can not only be heat insulation layer but also exposed in fire, so it is an ideal lining material for high temperature furnace. Today the bubble alumina ceramic has received more attention and become more and more important as the most promising high-temperature insulation material.

There are many factories producing bubble alumina ceramics in China. Most products have the properties of poor stability and low strength because of high melting points, strong ionic bonds and low diffusion coefficient of alumina and the low sintering temperature and unreasonable formula during production process. The scientists spend much time on the binders of alumina ceramics and modification of alumina bubbles in order to lower the sintering temperature, and improve the strength and quality uniform [9–12]. The results of different researchers have great differences. In this paper, the mechanical properties of alumina bubbles, the filling properties, the structure morphology and sintering theory were in deep studied based on existing studies on phosphoric acid combined bubble alumina ceramic and alumina bubble modification. The high strength bubble alumina ceramic was prepared with composite binders composed of ammonium alumina sulphate and phosphoric acid.

## 1. Experimental

### 1.1. Materials

Lightweight bubble alumina ceramics were prepared by alumina bubbles and $\alpha$-$Al_2O_3$ micro-powders. The chemical

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Table 1
Chemical composition of materials, %.

| Raw material | Component, % | | | | |
|---|---|---|---|---|---|
| | $Al_2O_3$ | $SiO_2$ | $Fe_2O_3$ | $Na_2O$ | Remark |
| Alumina bubble | ≥99 | 0.20 | 0.10 | | Particle size 0.2–4 mm |
| α-$Al_2O_3$ | ≥99 | 0.07 | 0.03 | 0.10 | $D_{50}$ is 6.99 μm |

Table 2
Composition of composite binders.

| No. | Ratio of ammonium aluminum sulphate solution and phosphoric acid solution | Weight of ammonium aluminum sulphate solution (A1), g | Weight of phosphoric acid solution (E1), g |
|---|---|---|---|
| F1 | 1:1 | 100 | 100 |
| F2 | 2:1 | 200 | 100 |
| F3 | 3:1 | 300 | 100 |
| F4 | 4:1 | 400 | 100 |
| F5 | 8:1 | 800 | 100 |

composition was shown in Table 1. Particle size of alumina bubbles was varied over a range of 0.2 mm to 4 mm. D50 of α-$Al_2O_3$ powders was 6.99 μm. Three kinds of binders were prepared, including diluted phosphoric acids with percentage concentration of 50% (E1), 25.0% (E2) and 12.5% (E3) by weight, ammonium aluminum sulphate solutions of 13.04% (A1) and 8.89% (A2), and composite binders (F) with the composition showed as Table 2.

### 1.2. Preparation of samples

The alumina bubbles were screened to four gradations according to FURNAS theory. The α-$Al_2O_3$ powders and alumina bubbles were well-mixed according to the composition as Table 3. After adding binders the ceramic body was formed by pressure vibration molding technique, then dried at 60 °C and demolded. After heat-treated at 1700 °C for 6 h the lightweight bubble alumina ceramic was prepared. The size of ceramic body was 40 mm × 40 mm × 50 mm and the density was 1.2 g/cm$^3$, 1.4 g/cm$^3$ and 1.6 g/cm$^3$ respectively. The weight percent of binders was 30–35% of powders.

Alumina bubbles with the size of 3–4 mm in range were immersed into binder solutions for 2 h, then dried at 80 °C for 12 h. After heated-treated at 900 °C, 1550 °C and 1700 °C for 2 h, the modified alumina bubbles were prepared.

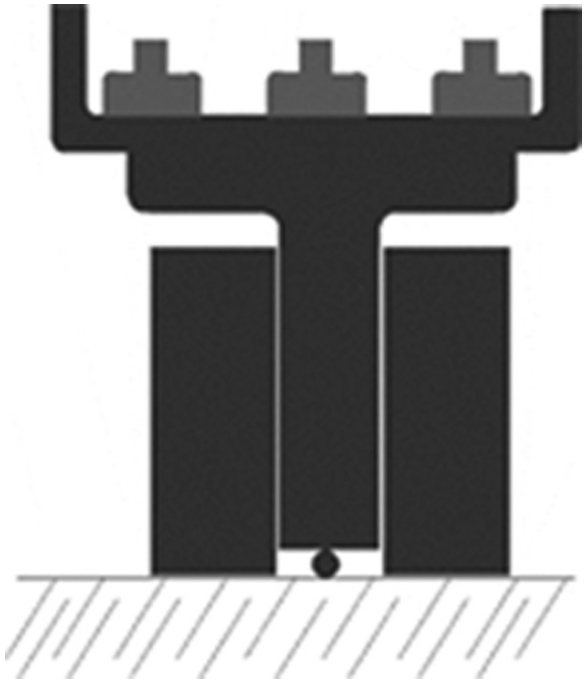

Fig. 1. Experimental device for measuring compressive resistance of alumina bubble.

### 1.3. Testing programs

The compressive strength of ceramic samples was determined by WE-B universal hydraulic testing machine. The micro-structure of the samples was analyzed using Hitachi s75 scanning electron microscopy (SEM). Compress resistance of single alumina bubble was examined with self-prepared equipment shown in Fig. 1.

## 2. Results and discussion

The compressive strength of lightweight bubble alumina ceramic combined by phosphoric acid solution and ammonium

Table 3
composition of lightweight bubble alumina ceramic.

| No. | Volume density, g/cm$^3$ | Size distribution of alumina bubble, wt% | | | | α-$Al_2O_3$ powder, wt% | Binders, wt% |
|---|---|---|---|---|---|---|---|
| | | 4–3 mm | 3–2 mm | 2–1 mm | 1–0.2 mm | | |
| G1 | 1.2 | 25 | 16 | 15 | 14 | 30 | 30–35 |
| G2 | 1.4 | 10 | 12 | 15 | 28 | 35 | |
| G3 | 1.6 | 4 | 7 | 10 | 39 | 40 | |

Table 4
compressive strength of lightweight bubble alumina ceramic combined by phosphoric acid solution and ammonium aluminum sulphate solution.

| No. | Binder | Concentration of binder | Compressive strength, MPa | | |
|---|---|---|---|---|---|
| | | | Density (1.2) | Density (1.4) | Density (1.6) |
| E1 | Phosphoric acid | 50% | 11.28 | 22.80 | 29.67 |
| E2 | | 25% | | 18.23 | |
| E3 | | 12.5% | | 12.72 | |
| A1 | Ammonium aluminum sulphate | 13.04% | 6.70 | 10.56 | 13.65 |
| A2 | | 8.89% | 5.76 | 8.32 | 10.40 |

aluminum sulphate solution was shown in Table 4. The compressive strength of alumina ceramic combined with ammonium alumina sulphate solution was lower than that combined with phosphoric acid solution. The compressive strength of alumina ceramic combined with A1 was 10.56 MPa, and combined with E1 achieved 22.80 MPa.

The ammonium alumina sulphate decomposed into nano γ-$Al_2O_3$ powders at 900 °C. These active γ-$Al_2O_3$ powders can promote the ceramic sintering. But the quantity of powders produced by ammonium alumina sulphate is so few that results in the low strength of ceramic. Phosphoric acid produces aluminum phosphate during sintering that due to the high strength of ceramic. Aluminum phosphate is a high-temperature binder and can form the transition layer on the surface of alumina bubble and micro-powders, which becomes $Al^{3+}$ source. The aluminum phosphate on surface becomes the diffusion channel of $Al^{3+}$ that accelerates the sintering of alumina bubbles contact neck resulting in the high strength [13].

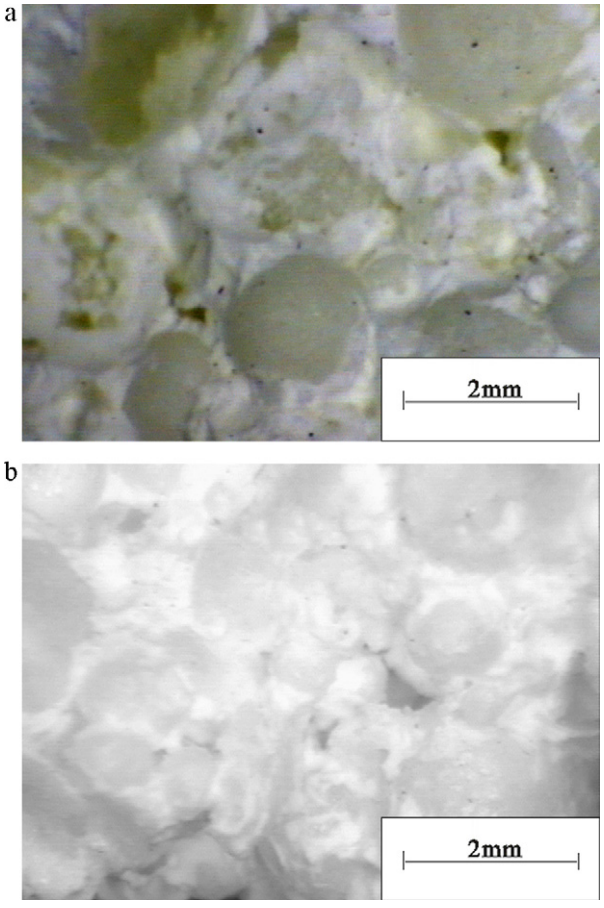


Fig. 2. Fracture morphology of bubble alumina ceramic combined with ammonium aluminum sulphate after thermal shock test (optical microscope).

Fig. 2 shows the fracture morphology of alumina ceramic combined with ammonium aluminum sulphate after thermal shock test (cycling 17 times at 1100 °C under air-cooling condition). The concentration of ammonium aluminum sulphate solution was 8.89% (A2) in Fig. 2(a) and 13.04% (A1) in Fig. 2(b). Along-ball fractures occurred in thermal shock test are shown in Fig. 2. The combining force between alumina bubbles and alumina powders is lower than the compressive strength of alumina bubbles with ammonium aluminum sulphate as a binder.

Fig. 3 shows the fracture morphology of alumina ceramic combined with phosphoric acid after thermal shock test (cycling 11–14 times). The concentration of phosphoric acid solution was 12.5% (E3) in Fig. 3(a), 25.0% (E2) in Fig. 3(b) and 50.0% (E1) in Fig. 3(c). The alumina bubbles are broken and trans-ball fractures emerge. The combining force between alumina bubbles and alumina powders is higher than the compressive strength of alumina bubbles with phosphoric acid as a binder.

The effect of binders on the compressive strength of alumina bubbles was studied. The results are shown in Table 5. The compressive strength of alumina bubbles modified by ammonium aluminum sulphate increased with the improving of sintering temperature. After heat-treatment at 900 °C the strength of ammonium aluminum sulphate was declined to 50% of that of unmodified alumina bubbles. When the temperature rose to 1500 °C, the compressive strength improved to 2 times of that of unmodified alumina bubbles, 1700 °C increased to 2.5 times. The compressive resistance of phosphoric acid modified alumina bubbles decreases as the temperature increases. After heat-treated at 1500 °C and 1700 °C, the strength of modified alumina bubble decreased to only half of that of unmodified alumina bubble, which resulted in the trans-ball fractures.

As shown in Tables 4 and 5, the compressive strength of alumina bubble modified by ammonium aluminum sulphate was improved, but that of bubble alumina ceramic combined with ammonium aluminum sulphate was decreased. That means the promoted sintering property of ammonium alumina sulphate was weak. The performance of phosphoric acid was opposite. The composite binder was prepared with ammonium aluminum sulphate solution (A1) and phosphoric acid solution

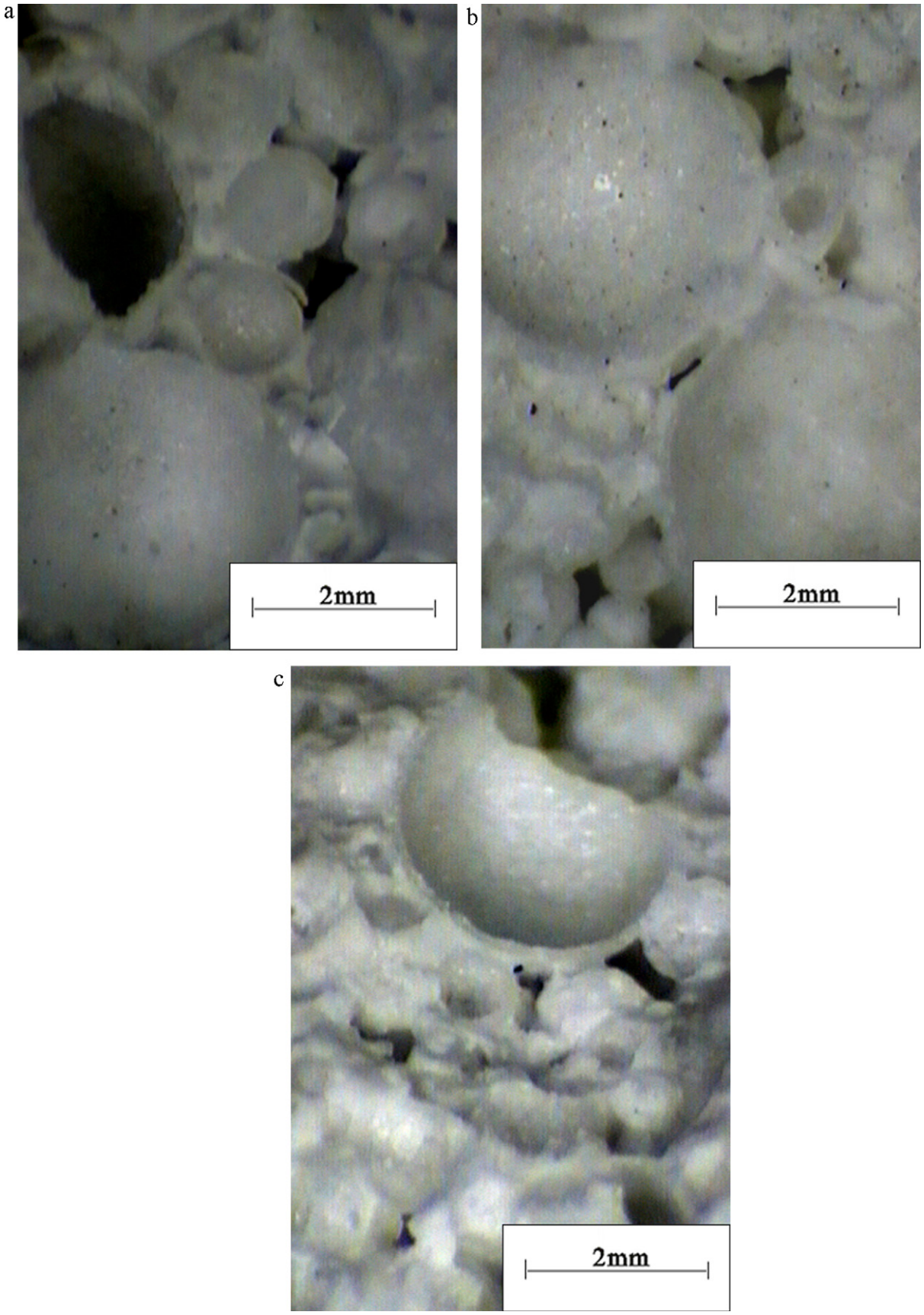


Fig. 3. Fracture morphology of bubble alumina ceramic combined with phosphoric acid after thermal shock test (optical microscope).

Table 5
Influence of different binders on pressure resistance of alumina bubble under different heat-treatment temperatures.

| Binder | Samples | | |
|---|---|---|---|
| | Modified Alumina bubble (heat-treatment at 900 °C for 2 h) | Modified Alumina bubble (heat-treatment at 1550 °C for 2 h) | Modified Alumina bubble (heat-treatment at 1700 °C for 2 h) |
| Without modification | 15.6 N | 16.0 N | 18.2 N |
| A1 | 7.65 N | 32.8 N | 38.7 N |
| A2 | 7.9 N | 30.1 N | 34.8 N |
| E1 | 20.3 N | 9.8 N | 9.7 N |
| E2 | 16.9 N | 9.5 N | 9.4 N |
| E3 | 14.7 N | 8.7 N | 8.7 N |
| F1 | 13.8 N | 13.2 N | 26.5 N |
| F2 | 11.7 N | 14.5 N | 28.6 N |
| F3 | 9.9 N | 17.6 N | 40.2 N |
| F4 | 9.7 N | 16.9 N | 38.4 N |
| F5 | 8.5 N | 25.9 N | 34.7 N |

(E1). The strength of alumina bubble modified by composite binder decreased with the increase of temperature for the low temperature range, but increased for the high temperature range. At 1700 °C, the strength improved to 2.5 times that of unmodified alumina bubble.

The compressive strength of composite binders with different compositions after heat-treatment at 1700 °C for 6 h was shown in Table 6. Compared to Table 4, composite binder can improve the compressive strength of bubble alumina ceramic effectively. The degree of improvement is related to the ratio of ammonium aluminum to phosphoric acid sulphate. The optimal ratio of A1 to E1 was 3:1(F3). The compressive strength of bubble alumina ceramic combined with F3 was 18.58–42.23 MPa, with the density of 1.2–1.6 g/cm$^3$. The effect of different composition of composite binder on the mechanical strength of bubble alumina ceramic was shown in Fig. 4, with the density of 1.4 g/cm$^3$. The compressive strength of alumina ceramic increased with the increase of ratio of E1. When A1:E1 equal to 3:1, the compressive strength achieved the largest value of 30.56 MPa. Then the strength decreased with the increase of ratio of A1. When A1:E1 equal to 1:1, the compressive strength achieved 18.95 MPa. Then the strength was improved as the increase of ratio of E1. The composite binder combined with the advantages of ammonium alumina sulphate and phosphoric acid and improved the compressive strength of both alumina bubbles and lightweight bubble alumina ceramic.

Table 6
Compressive strength of lightweight bubble alumina ceramic combined with composite binders.

| No. | Ratio of composite binder (A1:E1) | Compressive strength, MPa | | |
|---|---|---|---|---|
| | | Density (1.2) | Density (1.4) | Density (1.6) |
| F1 | 1:1 | | 18.95 | |
| F2 | 2:1 | | 24.08 | |
| F3 | 3:1 | 18.58 | 30.56 | 42.23 |
| F4 | 4:1 | | 22.72 | |
| F5 | 8:1 | | 13.66 | |

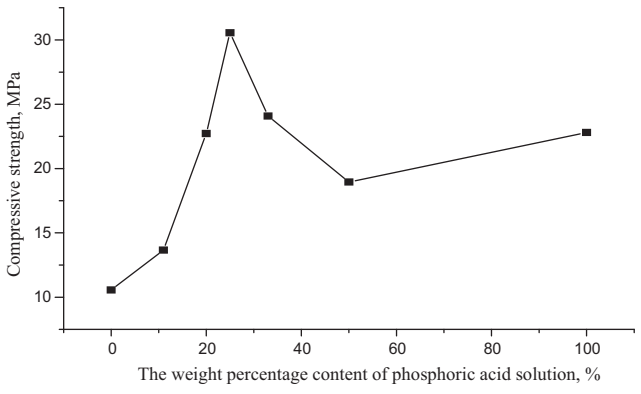


Fig. 4. Effect of the composition of composite binder on the mechanical property of lightweight bubble alumina ceramic.

## 3. Conclusions

Ammonium aluminum sulphate solution can improve compressive strength of alumina bubbles effectively but can not improve that of lightweight bubble alumina ceramic due to the fewer nano-alumina powders in situ decomposed of ammonium alumina sulphate. Trans-ball fractures occurred in thermal shock test. Phosphoric acid solution can improve compressive strength of alumina bubble ceramic due to promoting sintering properties of aluminum phosphate in situ produced by phosphoric acid and alumina component during sintering but decrease that of alumina bubbles. Along-ball fractures occurred in thermal shock test. The composite binder combined with the advantages of ammonium alumina sulphate and phosphoric acid and improved the compressive strength of both alumina bubbles and lightweight bubble alumina ceramic. The composite binder can reach excellent performance under low concentrations and effectively reduce the amount of the binders and lower the product cost. At the sintering temperature of 1700 °C, with composite ammonium alumina sulphate and phosphoric acid as the binder, the density of lightweight bubble alumina ceramic was between 1.20 and 1.60 g/cm$^3$, and the compressive strength was 18–42 MPa.

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