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Molten salt synthesis of α-Al_2O_3 platelets using $NaAlO_2$ as raw material

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Abstract

α-Al_2O_3 platelets were prepared by a molten salt synthesis method when $NaAlO_2$ was used as raw material. The effects of the stirring rate during the gel preparation, heating temperature, type and addition amount of molten salts, addition of plate-like α-Al_2O_3 seeds, additives such as $TiOSO_4$ and $Na_3PO_4 \cdot 12H_2O$ on the morphology of α-Al_2O_3 were studied. High stirring rate during the gel preparation and high heating temperature not only help to restrain the overlapping of α-Al_2O_3 platelets, but also improve the size distribution. When the heating temperature increases to 1200 °C, most of α-Al_2O_3 platelets are hexagonal in its morphology, and the size of platelets becomes relatively uniform. When Na_2SO_4–K_2SO_4 flux is used instead of NaCl–KCl flux, it is easy to obtain α-Al_2O_3 platelets with a big size. When the molar ratio of salt to final Al_2O_3 powders increases to 4:1, most of α-Al_2O_3 platelets are hexagonal, and the overlapping of powders is inhibited. The addition of a small amount of plate-like seeds has a significant effect on the size of α-Al_2O_3 platelets. With the increase of seed amount, the diameter of α-Al_2O_3 platelets tends to decrease. The addition of 5.45 wt.% $TiOSO_4$ results in the formation of hexagonal α-Al_2O_3 platelets with an average diameter of 5.1 μm and an average thickness of 1.4 μm. Thin α-Al_2O_3 platelets with a discal shape are obtained owing to the co-addition of 0.51 wt.% $Na_3PO_4 \cdot 12H_2O$ and 3 wt.% $TiOSO_4$.

Keywords: B. Platelets; α-Al_2O_3; Molten salt synthesis; Additives; Seeds

1. Introduction

α-Al_2O_3 is very important in ceramic industry because of the unique chemical, electrical and mechanical properties. Owing to its special shape, plate-like α-Al_2O_3 powders find some specific applications, such as the reinforcements in ceramic composites [1–3], the templates for the preparation of textured Al_2O_3 ceramics [4] and the fillers in the plastics to improve the thermal conductivity [5].

Molten salt synthesis is one of the most important methods to prepare plate-like α-Al_2O_3 powders. Compared with the conventional solid-state reaction, the temperature and time to synthesize plate-like α-Al_2O_3 in molten salts are greatly reduced. In the previous investigations, aluminum sulfate ($Al_2(SO_4)_3$) was often chosen as raw material [6–11]. The morphology of plate-like α-Al_2O_3 can be controlled by changing the heating temperature, heating time, molten salts, additives, seeds and so on [11]. However, it is deleterious to the environment due to the production of poisonous SO_3 and SO_2 during the thermal decomposition of $Al_2(SO_4)_3$. Plate-like α-Al_2O_3 single crystal particles were also prepared by a molten salt synthesis method using aluminum hydroxide ($Al(OH)_3$) as raw material [12]. From the viewpoint of environmental protection and economy, $Al(OH)_3$ may be superior to $Al_2(SO_4)_3$.

Sodium aluminate ($NaAlO_2$) is often used to synthesize $Al(OH)_3$. The process will be simplified if plate-like α-Al_2O_3 powders can be prepared directly from $NaAlO_2$. The present study aims to obtain α-Al_2O_3 platelets by a molten salt synthesis method when $NaAlO_2$ is chosen as raw material. The effects of the stirring rate during the gel preparation, heating temperature, type of molten salts and ratio of salt to powders on the morphology of α-Al_2O_3 were studied. Because the morphology of α-Al_2O_3 powders was also influenced by the addition of seeds [10,11] or additives such as Ti^{4+} and PO_4^{3-} [6,9,11], the effects of plate-like α-Al_2O_3 seeds, titanyl sulfate ($TiOSO_4$) and trisodium phosphate ($Na_3PO_4 \cdot 12H_2O$) on the morphology of α-Al_2O_3 platelets were also investigated.

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Table 1
The information of used materials in the experiment.

Raw material	Formula	Source	Purity
Sodium aluminum oxide	$NaAlO_2$	Zibo Tongjie Chemical Co., Ltd.	Industrial purity
Aluminum sulfate	$Al_2(SO_4)_3 \cdot 18H_2O$	Shanghai Meixing Chemical Co., Ltd.	99.5%
Sodium hydroxide	NaOH	Shanghai Xujiang Industrial Co., Ltd.	99.5%
Sodium carbonate	Na_2CO_3	Shanghai Hongguang Chemical Co., Ltd.	99.5%
Sodium chloride	NaCl	Shanghai Chemical Agent Co., Ltd.	99.5%
Potassium chloride	KCl	Shanghai Lingfeng Chemical Co., Ltd.	99.5%
Sodium sulfate	Na_2SO_4	Shanghai ShihuiHewei Chemical Co., Ltd.	99.5%
Potassium sulfate	K_2SO_4	Medical Combine of China, Shanghai Chemical Agent Company	99.0%
Trisodium phosphate	$Na_3PO_4 \cdot 12H_2O$	Medical Combine of China, Shanghai Chemical Agent Company	99.5%
Titanyl sulfate	$TiOSO_4$	The Fifth Company of Reagent, Shenyang	99.5%

2. Experimental procedure

$NaAlO_2$ (industrial purity, $M_{Al_2O_3}/M_{Na_2O} = 1.364$. Na_2O exists in the form of $NaAlO_2$, NaOH and Na_2CO_3), NaOH, $Al_2(SO_4)_3 \cdot 18H_2O$, Na_2CO_3, Na_2SO_4, K_2SO_4, NaCl and KCl were used. The information of used materials is shown in Table 1. Either mixture sulfate flux (Na_2SO_4:K_2SO_4 = 1:1, molar ratio) or mixture chloride flux (NaCl:KCl = 1:1, molar ratio) was used because the mixture salts were more beneficial to the development of α-Al_2O_3 platelets than pure salt.

α-Al_2O_3 platelets were prepared as follows: (1) $NaAlO_2$ and NaOH were dissolved in de-ionized water to make solution A, in which the concentration of Al_2O_3 was 53 g/L and pH = 14. (2) Solution B was obtained by dissolving $Al_2(SO_4)_3 \cdot 18H_2O$ in de-ionized water, and the concentration was 249 g/L. (3) Solution B was added slowly into solution A at room temperature and kept stirring rapidly for 18 min. The pH was adjusted to 11.0 by adding Na_2CO_3. The stirring rate was chosen to be 600, 900 and 1400 r/min, respectively. Finally $Al(OH)_3$ gel was obtained. (4) After $Al(OH)_3$ gel was leached for 10–15 min, Na_2SO_4–K_2SO_4 or NaCl–KCl was added. The addition amount of salt was calculated in terms of the resultant Al_2O_3. The molar ratio of salt to final Al_2O_3 powders was chosen to be 1:1, 2:1 and 4:1 separately. Then the mixtures were ball-milled in ethanol for 2 h using high-purity α-Al_2O_3 balls. (5) The semi-solid matters were placed in a covered alumina crucible, and then were heated for 3 h in air. The heating temperature was chosen to be 900, 1000, 1100 and 1200 °C, and the heating rate was 150 °C/h. (6) The reaction product was ultrasonic washed with de-ionized water repeatedly and then dried. Finally α-Al_2O_3 platelets were obtained. The flow chart of α-Al_2O_3 platelets is shown in Fig. 1.

To study the effect of α-Al_2O_3 seeds on the morphology of α-Al_2O_3 platelets, plate-like α-Al_2O_3 powders with an average diameter of 0.5 μm were added during the formation of solution B. The seed content was 0.1, 0.25, 0.5 and 1 wt.%, respectively. And also, in order to control the morphology of final α-Al_2O_3 powders, either 5.45 wt.% $TiOSO_4$ or 0.51 wt.% $Na_3PO_4 \cdot 12H_2O$ + 3 wt.% $TiOSO_4$ was added during the formation of solution B. The addition amount of $TiOSO_4$ and $Na_3PO_4 \cdot 12H_2O$ was presented by weight, respectively in terms of $TiOSO_4$ and P_2O_5, relative to the weight of final α-Al_2O_3 particles. The synthesis conditions are listed in Table 2.

The phase assembly of final powders was determined by X-ray diffraction analysis (XRD, D/MAX-RB, Rigaku Co., Japan). The morphology of α-Al_2O_3 particles was observed by scanning electron microscope (SEM, S-570, Hitachi Co., Japan). Dimensional statistics of α-Al_2O_3 platelets was collected by measuring SEM images of 150–200 particles.

3. Results and discussion

3.1. Effect of heating temperature on the morphology of α-Al_2O_3 platelets

XRD analysis and SEM observation indicate that α-Al_2O_3 platelets can be obtained by a molten salt synthesis method when $NaAlO_2$ is used as raw material. Fig. 2 presents the XRD patterns of the final powders, which are prepared according to the flow chart shown in Fig. 1. Even though the heating

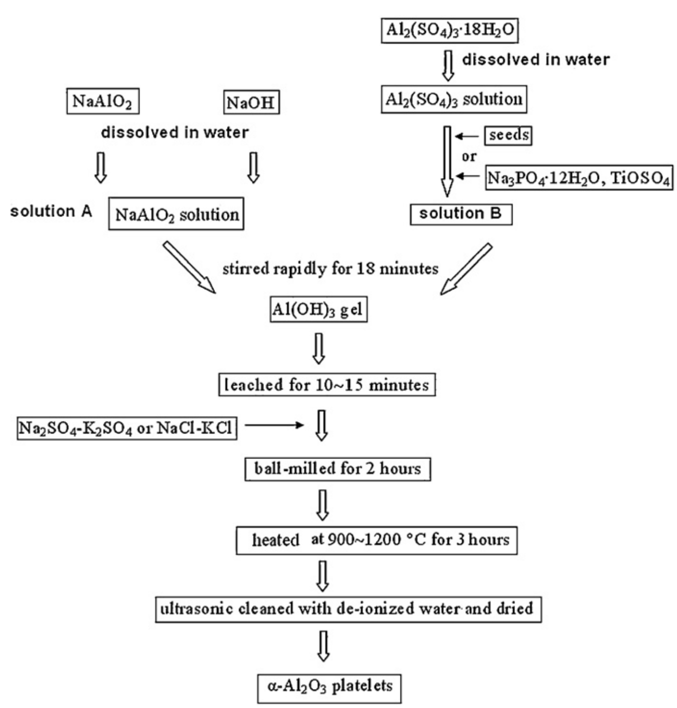

Fig. 1. Flow chart showing the synthesizing process of α-Al_2O_3 platelets.

Table 2
The synthesis conditions and size of α-Al_2O_3 platelets.

Sample	Heating temperature (°C)	Stirring rate (r/min)	Molten salts	The molar ratio of salt to powders	Seeds amount (wt.%)	Additives amount (wt.%)	Average diameter (μm)
A-1	900	1400	Na_2SO_4–K_2SO_4	2:1	–	–	5.3
A-2	1000						6.3
A-3	1100						6.5
A-4	1200						6.8
B-1	1200	600	Na_2SO_4–K_2SO_4	2:1	–	–	6.5
B-2		900					6.7
A-4		1400					6.8
C-1	1200	1400	Na_2SO_4-K_2SO_4	1:1	–	–	2.4
A-4				2:1			6.8
C-2				4:1			7.5
D-1	1200	1400	NaCl–KCl	4:1	–	–	5.8
C-2			Na_2SO_4–K_2SO_4				7.5
C-2	1200	1400	Na_2SO_4–K_2SO_4	4:1	0	–	7.5
E-1					0.1		3.2
E-2					0.25		2.5
E-3					0.5		1.9
E-4					1		1.5
D-1	1200	1400	NaCl–KCl	4:1	–	0	5.8
F-1						5.45 wt.% $TiOSO_4$	5.1
F-2						0.51 wt.% $Na_3PO_4·12H_2O$ + 3 wt.% $TiOSO_4$	5.5

temperature is as low as 900 °C, single phase α-Al_2O_3 can be synthesized.

The phase assembly is not affected by the increase of heating temperature, but the morphology and size of final α-Al_2O_3 platelets change appreciably. Fig. 3(a) shows the morphology of α-Al_2O_3 platelets synthesized at 900 °C. There are some overlapped particles, and the size distribution of α-Al_2O_3 platelets is broad. The increase of heating temperature is beneficial to improve the size distribution and overlapping. Most important of all, plate-like α-Al_2O_3 particles with regular shape are well developed. When the heating temperature increases to 1200 °C, most of α-Al_2O_3 platelets are hexagonal in its morphology, see Fig. 3(b). Not only the overlapping of α-Al_2O_3 platelets improves obviously, but also the size of platelets becomes relatively uniform.

When $Al_2(SO_4)_3$ solution is added slowly into $NaAlO_2$ solution, the following reactions will happen.

$$6NaAlO_2 + Al_2(SO_4)_3·18H_2O \rightarrow 8Al(OH)_3 + 3Na_2SO_4 + 6H_2O \quad (1)$$

$$6NaOH + Al_2(SO_4)_3·18H_2O \rightarrow 8Al(OH)_3 + 3Na_2SO_4 + 18H_2O \quad (2)$$

$$3Na_2CO_3 + Al_2(SO_4)_3·18H_2O \rightarrow 2Al(OH)_3 + 3Na_2SO_4 + 3CO_2 + 15H_2O \quad (3)$$

The formation of α-Al_2O_3 platelets in the molten salt should be a nucleation-growth process. When heated above 900 °C, the thermal decomposition of $Al(OH)_3$ results in the formation of α-Al_2O_3, which can act as the nuclei of α-Al_2O_3 platelets. In the later stage of platelet growth, some small α-Al_2O_3 platelets are consumed by the growth of large α-Al_2O_3 platelets through Oswald ripening. The increase of heating temperature is helpful to the development of α-Al_2O_3 platelets, and the size of α-Al_2O_3 platelets tends to be uniform owing to the disappearance of small particles.

3.2. Effect of stirring rate during the gel preparation on the morphology of α-Al_2O_3 platelets

Fig. 4(a)–(c) shows SEM micrographs of final α-Al_2O_3 platelets experienced in different stirring conditions. When the stirring rate is 600 r/min, there are a few well-developed

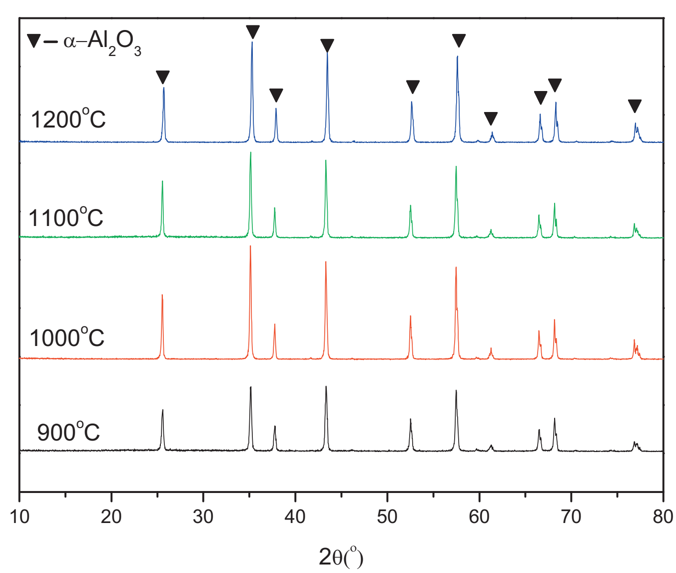

Fig. 2. The XRD patterns of the final powders heated at 900, 1000, 1100 and 1200 °C for 3 h.

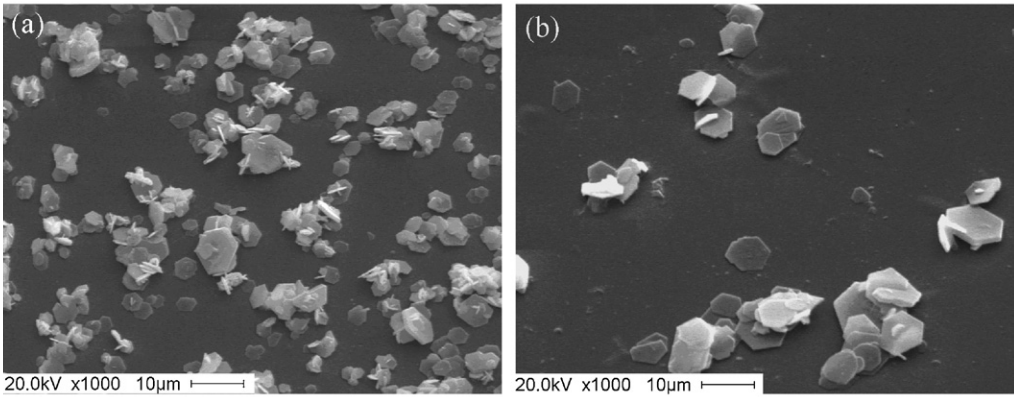

Fig. 3. The morphology of final α-Al_2O_3 platelets heated at different temperatures: (a) 900 °C and (b) 1200 °C.

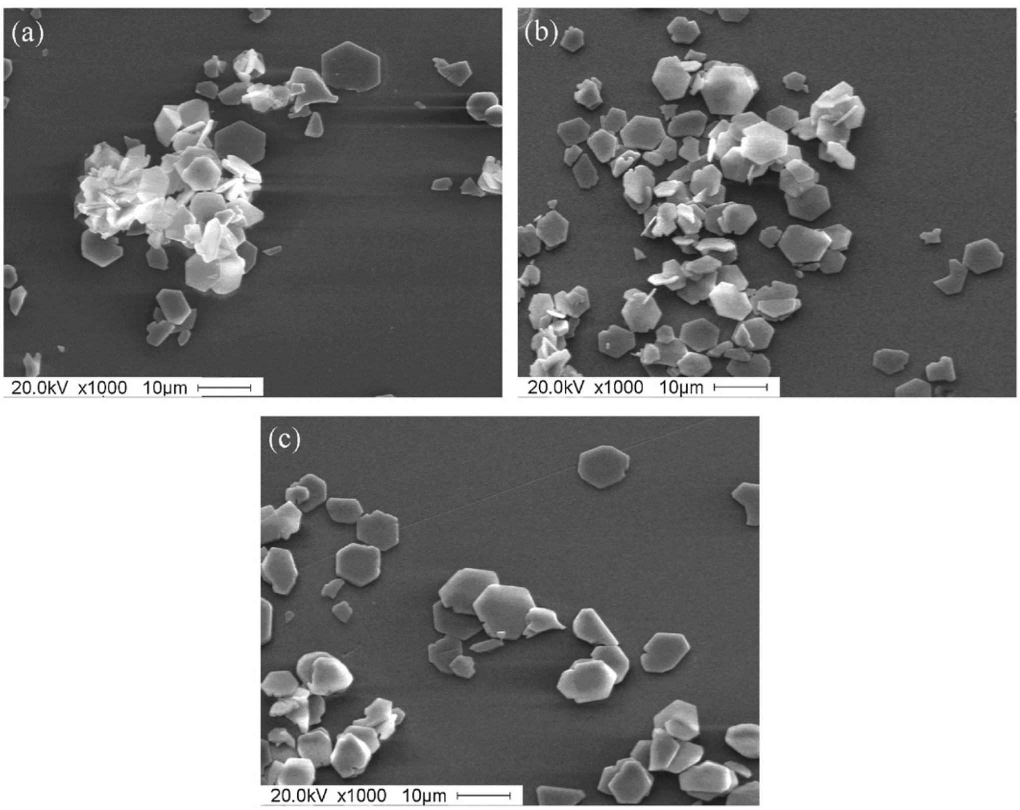

Fig. 4. The morphology of final α-Al_2O_3 platelets experienced in different stirring conditions: (a) 600 r/min; (b) 900 r/min and (c) 1400 r/min.

hexagonal α-Al_2O_3 platelets. The size distribution of α-Al_2O_3 platelets is very broad, and there are many overlapped particles. Under the stirring rate of 900 r/min, although the overlapping of α-Al_2O_3 platelets improves obviously, the size is still non-uniform. High stirring rate of 1400 r/min not only helps to restrain the overlapping of α-Al_2O_3 platelets, but also improves the size distribution.

It is easy to understand that the properties of product by $NaAlO_2$–$A1_2(SO_4)_3$ method are affected by the precipitation conditions, i.e., the reactant concentration, pH and temperature [13]. Our experimental results indicate that the stirring rate during the gel preparation is also a key factor to affect the morphology of α-Al_2O_3 platelets.

The reactivity of reaction (1)–(3) is relatively slow under low stirring rate, thus the uniformity of $Al(OH)_3$ gel is not very well. In the following ball-milling, it is difficult to obtain a mixture containing $Al(OH)_3$ precursor with a very homogeneous distribution of molten salts. Correspondingly, the non-uniformity of α-Al_2O_3 nucleation sites has a bad influence upon the crystal growth of platelets. Thus under the stirring rate of 600 r/min, α-Al_2O_3 platelets have a wide size distribution, and there are many overlapped particles. When the stirring rate increases to 1400 r/min, it is relatively easy to disperse the molten salts very homogeneously in $Al(OH)_3$ precursor by ball-milling, α-Al_2O_3 crystals tend to grow uniformly in size. The size distribution of α-Al_2O_3 platelets is narrow, and the overlapping is inhibited effectively.

3.3. Effect of molten salts on the morphology of α-Al_2O_3 platelets

In addition to the stirring rate, the morphology of final α-Al_2O_3 platelets is affected by molten salts. Fig. 5(a) shows the morphology of α-Al_2O_3 powders synthesized in Na_2SO_4–K_2SO_4 flux when the molar ratio of salt to final Al_2O_3 powders is 1:1. The shape of α-Al_2O_3 particles is irregular, and their size is small. The use of more molten salts brings about bigger crystal growth space and higher diffusivity of components in the molten salts. When the molar ratio of salt to Al_2O_3 powders increases to 2:1, some hexagonal α-Al_2O_3 platelets can be obtained, but there are some overlapped particles, as shown in Fig. 5(b). The molar ratio of salt to Al_2O_3 powders further increases to 4:1, most of α-Al_2O_3 platelets are hexagonal in its morphology, and the overlapping of powders is inhibited, see Fig. 5(c). When NaCl–KCl is used, though the molar ratio of salt to Al_2O_3 powders is still kept to be 4:1, the size of α-Al_2O_3 platelets becomes small and the overlapping becomes serious, as shown in Fig. 5(d). Because sulfates have greater ionic strength, higher solubility is ensured, and α-Al_2O_3 platelets can be developed in sulfates better than in chlorides. Therefore, α-Al_2O_3 platelets with a big size are obtained when sulfates are used instead of chlorides.

3.4. Effect of plate-like seeds on the morphology of α-Al_2O_3 platelets

The addition of a small amount of seeds has a significant effect on the size of α-Al_2O_3 platelets. Fig. 6 shows SEM micrographs of final α-Al_2O_3 platelets synthesized in Na_2SO_4–K_2SO_4 flux when 0.1 wt.%, 0.5 wt.% and 1 wt.% seeds are added. For convenience to compare, the micrograph of α-Al_2O_3 platelets without the addition of seeds is also given. The average diameter of α-Al_2O_3 platelets decreases from about 7.5 μm to 3.2 μm quickly even though added by 0.1 wt.% plate-like seeds. With the increase of seed amount, the diameter of α-Al_2O_3 platelets tends to decrease. The variation of average diameter of final α-Al_2O_3 platelets with the amount of α-Al_2O_3 seed added is shown in Fig. 7. When 1 wt.% seeds are added, the average diameter decreases to 1.5 μm.

When heated at 1200 °C, α-Al_2O_3 resulting from the thermal decomposition of $Al(OH)_3$ acts as the nuclei of α-Al_2O_3 platelets. When α-Al_2O_3 seeds are added, extra nucleation sites can be provided by the seeds since they have the same crystal structure as α-Al_2O_3 nuclei and their size is small. Then α-Al_2O_3 particles grow large from the nuclei provided by both plate-like seeds and α-Al_2O_3 formed by the decomposition of $Al(OH)_3$. Thus fine α-Al_2O_3 platelets are obtained accompanied by the increase in the nuclei of α-Al_2O_3. Moreover, the

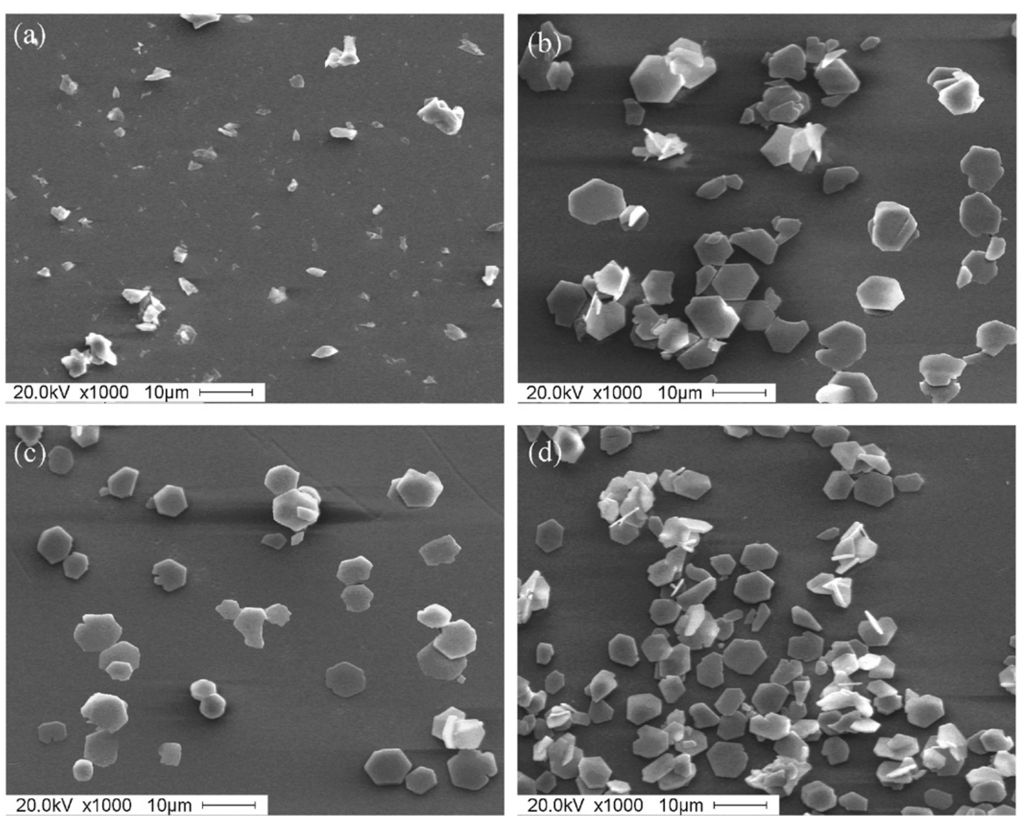

Fig. 5. The morphology of final α-Al_2O_3 platelets prepared using different molten salts and different ratio of salt to Al_2O_3 powders: (a) Na_2SO_4–K_2SO_4, ratio of salt to powders 1:1; (b) Na_2SO_4–K_2SO_4, ratio of salt to powders 2:1; (c) Na_2SO_4–K_2SO_4, ratio of salt to powders 4:1 and (d) NaCl–KCl, ratio of salt to powders 4:1.

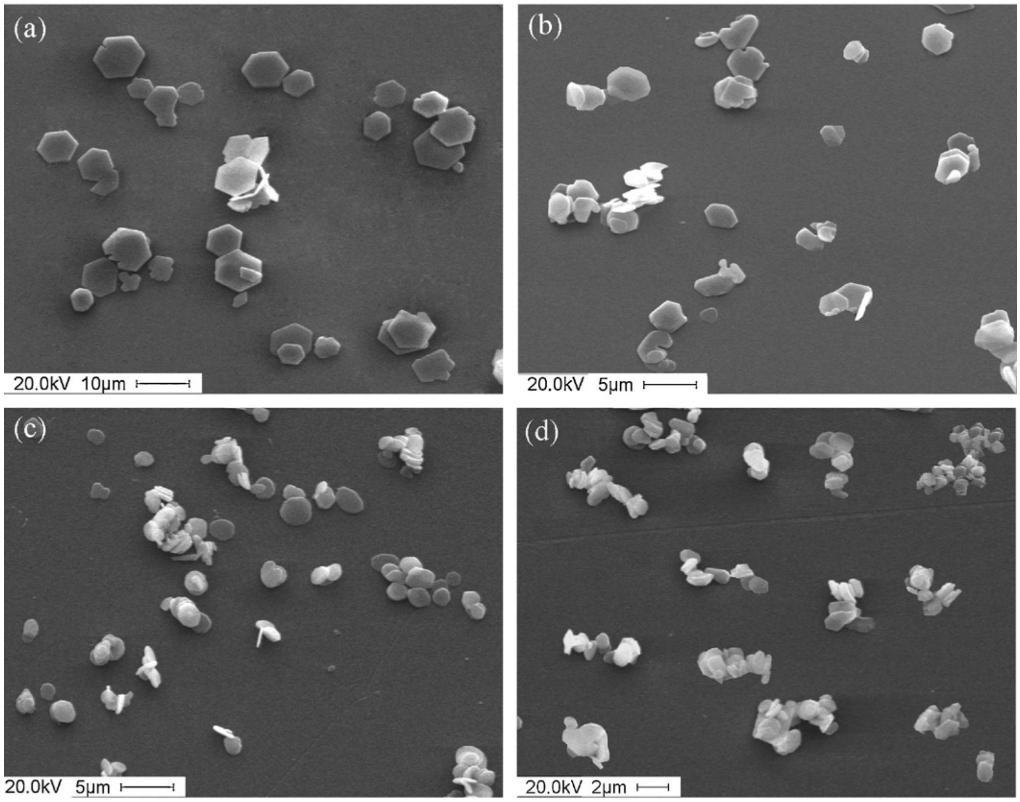

Fig. 6. The morphology of final α-Al_2O_3 platelets prepared by adding different amount of seeds: (a) 0 wt.% seeds; (b) 0.1 wt.% seeds; (c) 0.5 wt.% seeds and (d) 1 wt.% seeds.

diameter of final α-Al_2O_3 platelets decreases with the increase in the addition amount of plate-like α-Al_2O_3 seeds.

3.5. Effect of additives on the morphology of α-Al_2O_3 platelets

The morphology of α-Al_2O_3 platelets is easily controlled by the additives. By comparing Fig. 8(b) with Fig. 8(a), it is found that the addition of $TiOSO_4$ is helpful in the formation of hexagonal α-Al_2O_3 platelets. Meanwhile, α-Al_2O_3 particles tend to become small and thick. When 5.45 wt.% $TiOSO_4$ is added, the average diameter of final α-Al_2O_3 platelets decreases slightly to 5.1 μm, and the average thickness increases quickly to 1.4 μm. The co-addition of 0.51 wt.% $Na_3PO_4 \cdot 12H_2O$ and 3 wt.% $TiOSO_4$ makes it possible to form thin α-Al_2O_3 platelets with a discal shape, see Fig. 8(c). In comparison with Fig. 8(b), there is a slight increase in the diameter of α-Al_2O_3 platelets, and the thickness is reduced to 0.9 μm. At the same time, the overlapping of α-Al_2O_3 platelets is restrained obviously even though synthesized in NaCl–KCl flux.

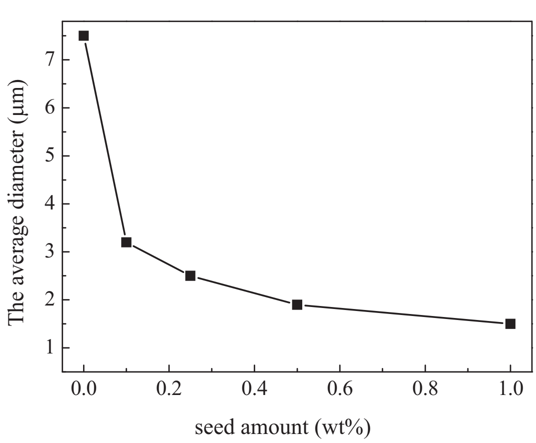

Fig. 7. The variation of average diameter of final α-Al_2O_3 platelets with the amount of α-Al_2O_3 seed added.

The crystal development is regarded as a ''growth unit'' course. According to the theoretical model of anionic coordination polyhedron growth units, the crystal growth and final morphology rely on the crystallographic orientation and combination manner of growth units [14]. In molten salts, the growth unit of α-Al_2O_3 should be [Al–O_6] octahedron. $\{0\,0\,0\,1\}$ faces appear predominantly, $\{1\ 0\ \bar{1}\ 0\}$ faces usually disappear and $\{1\ 1\ \bar{2}\ 0\}$ faces sometimes appear. Therefore, it is possible to obtain α-Al_2O_3 platelets with a hexagonal shape.

Titanium salt such as $TiOSO_4$ was chosen by Nitta et al. to change the shape of the synthesized Al_2O_3 [6,11]. The chemical analysis was done by Nitta, showing that flaky Al_2O_3 contains nearly as much titanium as the starting material [6]. It indicates that it is possible for Ti^{4+} ions to diffuse into the crystal lattice of Al_2O_3 at high temperatures, though the driven force is not clear. In order to keep the electrostatic balance, three Ti^{4+} ions substitute four Al^{3+} ions. Owing to the existence of extra Al^{3+} vacancies, the difference in the growth velocity of various

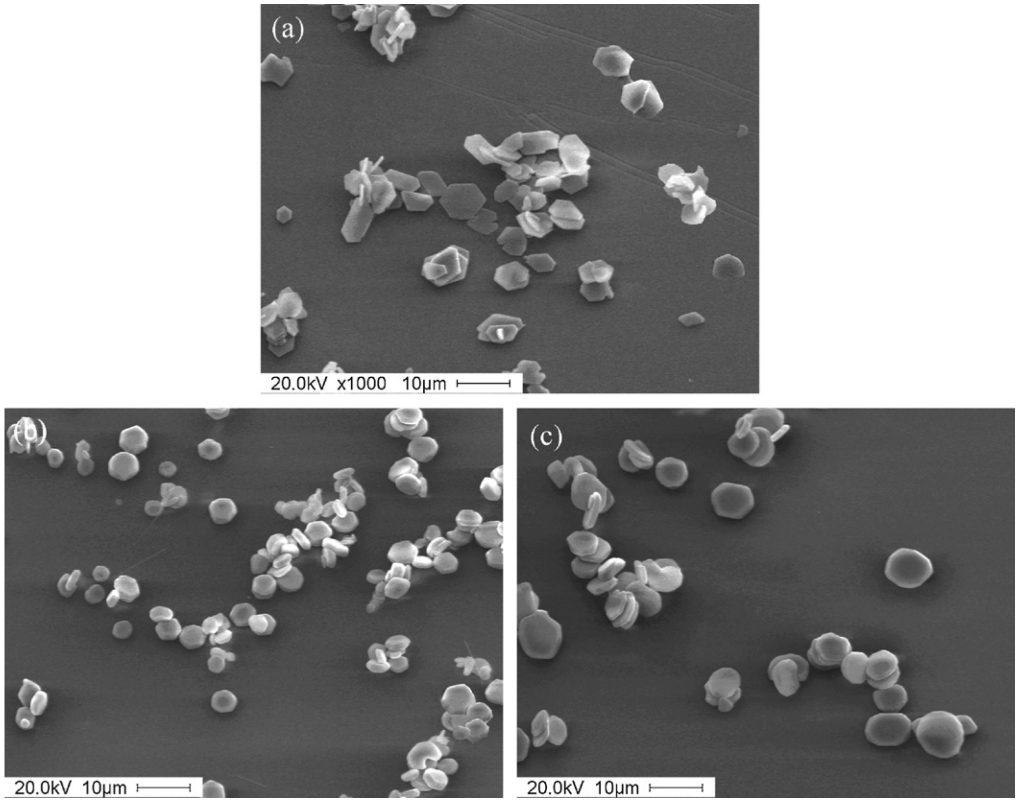

Fig. 8. The morphology of final α-Al_2O_3 platelets prepared in NaCl–KCl flux and with varied additives: (a) without additives; (b) with 5.45 wt.% $TiOSO_4$ and (c) with 0.51 wt.% $Na_3PO_4 \cdot 12H_2O$ + 3 wt.% $TiOSO_4$.

crystal faces may be reduced. Thus the addition of $TiOSO_4$ helps to obtain well-developed hexagonal α-Al_2O_3 particles with increased thickness. However, when $Na_3PO_4 \cdot 12H_2O$ is added, the superimposition of growth units on {0 0 0 1} faces is effectively prohibited by PO_4^{3-}, and the growth of α-Al_2O_3 along the thickness direction is restricted. Thin α-Al_2O_3 platelets with a discal shape are obtained under the combination action of PO_4^{3-} and Ti^{4+}.

4. Conclusions

1. When heated above 900 °C, the single phase α-Al_2O_3 platelets can be obtained by a molten salt synthesis method using $NaAlO_2$ as raw material. The increase of heating temperature is beneficial to the development of α-Al_2O_3 platelets. And also, the size distribution and the overlapping of α-Al_2O_3 platelets are improved. When the heating temperature increases to 1200 °C, most of α-Al_2O_3 platelets are hexagonal in its morphology, and the particle size becomes relatively uniform.
2. The stirring rate during the gel preparation is a key factor to affect the morphology of α-Al_2O_3 platelets. High stirring rate not only helps to restrain the overlapping of α-Al_2O_3 platelets, but also improves the size distribution.
3. The morphology of α-Al_2O_3 platelets is also affected by the type of molten salts used. When Na_2SO_4–K_2SO_4 flux is used instead of NaCl–KCl flux, it is easy to obtain α-Al_2O_3 platelets with a big size. When the molar ratio of Na_2SO_4–K_2SO_4 flux to final Al_2O_3 powders increases to 4:1, most of α-Al_2O_3 platelets are hexagonal, and the overlapping of powders is inhibited.
4. The addition of a small amount of plate-like seeds has a significant effect on the size of α-Al_2O_3 platelets. With the increase of seed amount, the diameter of α-Al_2O_3 platelets tends to decrease.
5. The morphology of α-Al_2O_3 platelets is easily controlled by the additives. The addition of 5.45 wt.% $TiOSO_4$ results in the formation of hexagonal α-Al_2O_3 platelets with an average diameter of 5.1 μm and an average thickness of 1.4 μm. Thin α-Al_2O_3 platelets with a discal shape are obtained owing to the co-addition of 0.51 wt.% $Na_3PO_4 \cdot 12H_2O$ and 3 wt.% $TiOSO_4$.

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