



Morphology-controlled synthesis of α -alumina microplatelets through an additive-assisted molten salt reaction



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ABSTRACT

Highly dispersed α -Al₂O₃ microplatelets of various sizes and aspect ratios were prepared by additive-assisted molten salt synthesis. The effects of SiO₂ and SiO₂ + CaO additions on phase formation and morphology development of α -Al₂O₃ particles were investigated. Introducing additives are very effective in controlling α -Al₂O₃ morphology. SiO₂ additive significantly enhances shape anisotropy of α -Al₂O₃ platelets, where a particle aspect ratio \sim 23.8 was achieved, about 2.7–6.0 times higher than those of the platelets prepared without additives. When using SiO₂ + CaO mixture, low concentration additive facilitates the growth of large platelets (\sim 9.8 μ m), while increasing its content dramatically reduces the platelet size to \sim 3.7 μ m. These findings provide a new insight for the design and synthesis of novel advanced anisotropic microcrystals with improved quality.

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1. Introduction

α -Al₂O₃ platelets have attracted extensive attention recently due to their unique mechanical, electrical, thermal and chemical properties. Because of the special two-dimensional structure, they are widely used as reinforcements in composites to enhance mechanical properties [1–3], as templates for fabricating textured ceramics with anisotropic properties [4,5], and as fillers to plastics for thermal conductivity improvement [6] etc. Since the applications of α -Al₂O₃ particles strongly depend on their morphologies [1–7], controlling morphological characteristics of α -Al₂O₃ are essential.

Molten salt synthesis (MSS) is a very important method to fabricate anisotropic microcrystals [8,9]. Different from solid-state reaction, the unconstrained growth environment in molten salt (s) facilitates formation of less/non-aggregated platelets according to α -Al₂O₃ crystal structure. Among the factors [9–13] that affect the morphology of α -Al₂O₃ particles, introducing additives play a positive role in controlling crystalline growth habits in molten

salt(s). As the well-known additives, PO₄^{3−} and Ti⁴⁺ are very helpful to modify the morphology of α -Al₂O₃ platelets [10,13]. Unfortunately, these additives either substantially reduce aspect ratio or remarkably broaden size distribution range of the products [10,13], which are not beneficial to significantly improve structural/functional properties of α -Al₂O₃ applications.

SiO₂-containing additives (either SiO₂ or SiO₂ + CaO) favor anisotropic grain growth inside alumina ceramics through forming liquid phase(s) at grain boundaries [4,14]. This merit makes them to be promising morphological modifiers for α -Al₂O₃ particles during MSS. However, the effects of SiO₂ and CaO additives on crystallization behavior and morphology characteristics of α -Al₂O₃ particles have been rarely studied. In this work, we report our achievements on the above-mentioned issues.

2. Experimental procedures

Equiaxed γ -Al₂O₃ particles (\sim 60 nm) were used as the precursor, and SiO₂ (\sim 15 nm) and CaCO₃ (\leq 100 nm) were used to provide SiO₂ and CaO + SiO₂ (1:1, molar ratio) additives during MSS. For salt effect study, γ -Al₂O₃ was mixed with Na₂SO₄ or Na₂SO₄ + K₂SO₄ mixture (1:1, molar ratio) at different molar ratios. For additive effect study, different amounts of SiO₂ and SiO₂ + CaCO₃ were added to γ -Al₂O₃ + Na₂SO₄ + K₂SO₄ mixture (1:1:1, molar ratio).

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All powder mixtures were ball-milled in ethanol for 24 h. After drying, they were heated at 5 °C/min to 1100–1200 °C and held for 1–4 h. The final products were washed and separated with hot deionized water several times to remove the salt(s) and residual additive(s). Crystalline phase was determined by X-ray diffraction (XRD, D/max 2400, Rigaku Corporation, Tokyo, Japan). Morphological features were observed by field-emission scanning electron microscopy (FE-SEM, HELIOS NanoLab 600i, FEI Corporation, OR, USA).

3. Results and discussion

Fig. 1 shows SEM micrographs of Al_2O_3 particles synthesized (a) without salts and with (b) Na_2SO_4 or (c, d) $\text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$ salt(s). Spherical particles with an average diameter (d) $\sim 0.2 \mu\text{m}$ were obtained without salts at 1100 °C. According to the XRD analysis, they are mainly of $\gamma\text{-Al}_2\text{O}_3$ phase. Adding Na_2SO_4 and $\text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$ salts accelerated $\alpha\text{-Al}_2\text{O}_3$ phase formation, producing pure

$\alpha\text{-Al}_2\text{O}_3$ at 1100 °C. Well-dispersed hexagonal platelets with $d \sim 3.2 \mu\text{m}$ and diameter/thickness aspect ratio (d/t) ~ 6.2 (Fig. 1e) were obtained from $\gamma\text{-Al}_2\text{O}_3 + 3\text{Na}_2\text{SO}_4$. Mixture $\gamma\text{-Al}_2\text{O}_3 + \text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$ yielded larger platelets with $d \sim 5.7 \mu\text{m}$ and $d/t \sim 8.7$. Doubling $\text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$ amount leads to slight decreases of both d and d/t . For the oriented platelets synthesized from $\gamma\text{-Al}_2\text{O}_3 + \text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$, the XRD pattern (Fig. 1f) is dominated by 0006 and 00012 peaks, confirming that their basal faces are of {0 0 0 l } planes.

Fig. 2 presents XRD patterns of Al_2O_3 particles synthesized with (a) $x \text{ wt\% SiO}_2$ and (b) $x \text{ wt\% SiO}_2 + y \text{ wt\% CaO}$ (1:1, molar ratio) additives. For the particles formed with SiO_2 and heated at 1150 °C, pure $\alpha\text{-Al}_2\text{O}_3$ was achieved at $x = 0$ and 0.15, but $\gamma\text{-Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$ coexisted at $x = 0.30$ and 0.50. Transformation of $\gamma\text{-Al}_2\text{O}_3$ to $\alpha\text{-Al}_2\text{O}_3$ completed at 1200 °C for $x = 0.30$ and 0.50, suggesting that more SiO_2 retards $\alpha\text{-Al}_2\text{O}_3$ phase transition and crystallization. For the particles synthesized with $\text{CaO} + \text{SiO}_2$ and heated at 1150 °C, $\gamma\text{-Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$ coexisted at $x = 0.155$ and

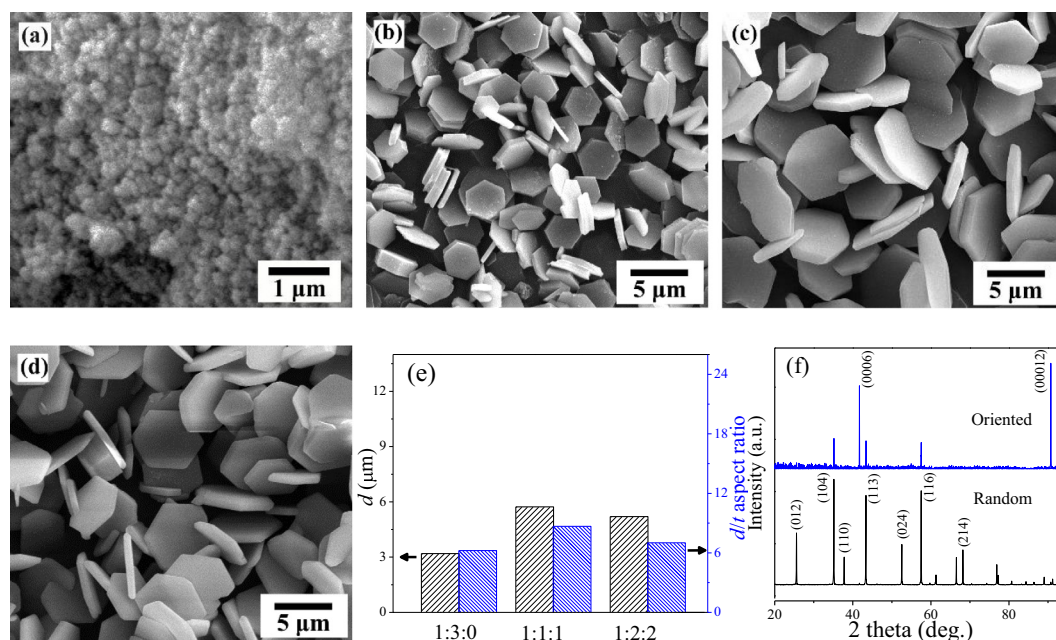


Fig. 1. SEM micrographs of Al_2O_3 particles synthesized with $\gamma\text{-Al}_2\text{O}_3/\text{Na}_2\text{SO}_4/\text{K}_2\text{SO}_4 =$ (a) 1:0:0, (b) 1:3:0, (c) 1:1:1 and (d) 1:2:2 at 1100 °C, respectively; (e) Average diameter and diameter/thickness aspect ratio of $\alpha\text{-Al}_2\text{O}_3$ platelets; (f) XRD patterns of $\alpha\text{-Al}_2\text{O}_3$ platelets synthesized from $\gamma\text{-Al}_2\text{O}_3 + \text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$ mixture: randomly oriented platelets and well-oriented ones aligned by casting these platelets on a glass substrate.

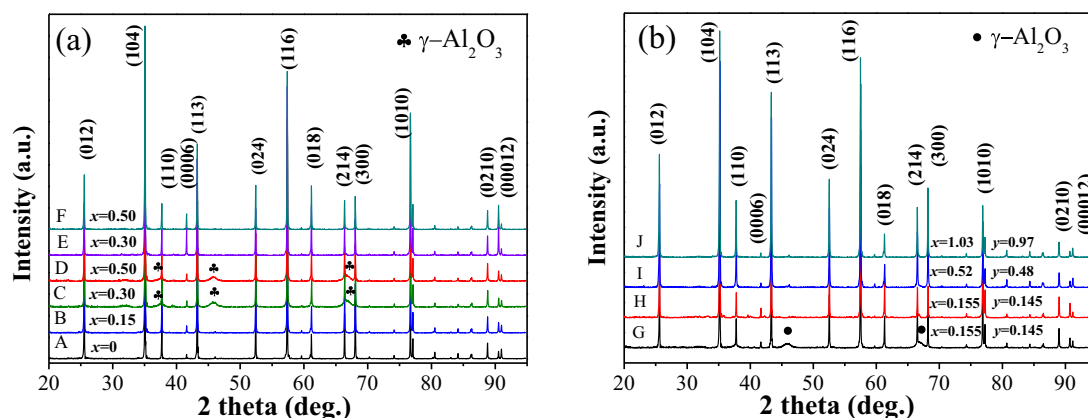


Fig. 2. XRD patterns of Al_2O_3 particles synthesized with (a) $x \text{ wt\% SiO}_2$ and (b) $x \text{ wt\% SiO}_2 + y \text{ wt\% CaO}$ at 1150 °C (A–D, G, I, and J) or 1200 °C (E, F and H), respectively.

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