Chemical synthesis of SmAlO3 by stearic acid route: structure, morphology and microwave dielectric properties

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Abstract
A rapid and facile approach was developed for the synthesis of ultrafine SmAlO3 powders through spontaneously combusting stearic acid precursors. The obtained products were characterized by typical techniques including X-ray diffraction (XRD), Fourier Transform Infrared (FT-IR), thermogravimetric and differential thermal analysis (TG-DTA), scanning electron microscopy (SEM) and electron microscopy transmission (TEM) to analyze the phase composition and microstructure. The dielectric characteristics of SmAlO3 microwave ceramics using the as-obtained products as original materials were also studied. Comparing with conventional solid-state reaction method, the synthesis temperature was dramatically reduced to 750 °C. Large size sheet structure was composed of a number of micro/nano-scale crystallites that were almost irregular in shape due to the mutual growth and overlapped shape of adjacent grains. The SmAlO3 ceramics with high density and uniform microstructure were obtained after sintering at 1500 °C for 4 h due to good sintering activity of the as-synthesized powders. In addition, desired dielectric properties at microwave frequencies (dielectric constant \( \varepsilon_r = 20.22 \), quality factor \( Q \cdot f = 74110 \) GHz, and temperature coefficient of resonant frequency \( TC_f = -74.6 \) ppm/°C) were achieved.

1. Introduction
In the high-frequency region, microwave dielectric ceramics are regarded as suitable materials for application in modern communication systems because they exhibit good properties, such as low \( \varepsilon_r \), high \( Q \cdot f \) and near-zero \( TC_f \) [1-4]. The rare-earth aluminate ceramics with perovskite structure are such materials and therefore have attracted many interests owing to their good dielectric properties in recent decades. Cho et al. reported the \( \varepsilon_r \) and \( Q \cdot f \) values of 20.4 and 65000 GHz for SmAlO3 ceramics prepared by solid state reaction method, indicating potential application in wireless communication used as dielectric resonators, filters as well as dielectric antenna [5]. The high dielectric constant and \( Q \cdot f \) can miniaturize the size and ensure a good frequency selectivity of these microwave devices. The large and negative \( TC_f \) of SmAlO3 ceramics, which is a shortcoming for practical applications, can be easily adjusted to a near-zero value by ion doping or constructing multiphase materials. A variety of novel SmAlO3-based ceramic systems have been explored in recent
years. For example, Suvorov et al. reported a new ceramics system with satisfactory microwave dielectric properties ($\varepsilon_r = 41$, $Q\cdot f = 42,000$ GHz, $T_C f = - 18$ ppm/°C) by mixing $0.65\text{CaTiO}_3$ and $0.35\text{SmAlO}_3$ at 1450 °C [6]. Soon afterwards, a similar competitive composition of $0.66\text{CaTiO}_3 - 0.34\text{SmAlO}_3$ in this system was found by Xu et al., displaying the best $\varepsilon_r$ and $Q\cdot f$ of 42.49 and 46069.86 GHz when sintered at 1500 °C for 4 h [7]. The comprehensive dielectric properties with $\varepsilon_r = 58.3$, $Q\cdot f = 18800$ GHz and $T_C f = 2.3$ ppm/°C could be obtained in the ceramic $0.75(Sr_{0.3}Ca_{0.427}Nd_{0.182})\text{TiO}_3 - 0.25\text{SmAlO}_3$ after being sintered at 1500 °C for 4 h [8]. Solomon revealed that the $0.1\text{SmAlO}_3-0.9\text{Ba(Zn}_{1/2}\text{Nb}_{2/3})\text{O}_3$ composite sintered at 1400 °C for 4 h had $\varepsilon_r = 37$, $T_C f = 25$ ppm/°C and high $Q\cdot f$ [9]. Moreover, the composite could find a potential application in optoelectronics communication. In addition, improved electrical properties were achieved by doping a small amount of $\text{SmAlO}_3$ to $(K_{0.44}\text{Na}_{0.52}\text{Li}_{0.04})(\text{Nb}_{0.91}\text{Ta}_{0.05}\text{Sb}_{0.04})\text{O}_3$ lead-free ceramics [10]. Based on these attempts, it is concluded that $\text{SmAlO}_3$ is one of the most competitive temperature compensation materials due to its rather large and negative $T_C f$.

Nevertheless, an outstanding drawback namely ultrahigh sintering temperature still prevents wide scale industrial application in microwave components for $\text{SmAlO}_3$-based ceramics owing to the poor reactive nature of the powders obtained from the conventional solid state reaction method. Moreover, the conventional solid state reaction method generally requires a prolonged preparation cycle and thus results in compositional deviation and particle coarsening of final products as well as excessive energy consumption. Especially, $\text{SmAlO}_3$ ceramics prepared by solid state reaction route require very high calcination and sintering temperatures of about 1400 °C and 1650 °C to be densified, respectively [11]. Recently, tremendous efforts have been devoted to enhancing the sintering performance and a variety of synthesis approaches have been carried out, such as co-precipitation method, molten salt synthesis, hydrothermal synthesis, sol-gel route and polymer complex method [12-17]. These wet-chemistry methods show several advantages like lower synthesis temperature, shorter reaction time and finer grain size, which influence the sintering and dielectric properties.
positively.

In our previous work, we investigated the synthesis of SmAlO$_3$ powders by citrate precursor and EDTA precursor methods [18, 19], exhibiting specific advantages in the reduced sintering temperature and enhanced microwave dielectric properties. Providing an important supplement of wet-chemistry methods for SmAlO$_3$ ceramics, we synthesized pure phase SmAlO$_3$ nanosized powders with high reaction yield and synthetic reproducibility by stearic acid method, in which stearic acid was used as not only solvent but also complexing agent. The method can also show a cost advantage due to the utilization of inexpensive metal salts and stearic acid as starting materials. The phase composition, microstructure and morphology of the obtained nanoparticles were analyzed and the microwave dielectric properties of the ceramics made by the nanoparticles were also discussed.

2. Experimental Procedures
2.1 Powder synthesis
SmAlO$_3$ powders were synthesized according to a synthetic procedure as shown in Fig. 1. In the experiments, analytical grade reagents samarium oxide (Sm$_2$O$_3$), aluminum nitrate hexahydrate [Al(NO$_3$)$_3$·9H$_2$O] and stearic acid (C$_{17}$H$_{35}$COOH) were chosen as raw materials. In light of its hygroscopic nature, Sm$_2$O$_3$ powder must be prefired at 900 °C for 4 h before the weighing procedure. First of all, 0.005 mol Sm$_2$O$_3$ was dissolved in diluted nitric acid to form a transparent solution under magnetic stirring at 80 °C. In addition, 30 g stearic acid was weighted and then heated slowly to melt completely at 75 °C in a beaker, then 0.01 mol Al(NO$_3$)$_3$·9H$_2$O was slowly added and dissolved in the molten stearic acid until obtained a clear solution with continuous stirring. Next, the Sm(NO$_3$)$_3$ solution was added dropwise to the molten stearic acid in the same way. By increasing the heating temperature to 100 °C for a sufficient period of time, the unnecessary water in the mixture was removed. After that, the mixture was continuously magnetic stirred and maintained at 130 °C until a uniform sol-like solution was produced, accompanied by the release of a large volume of reddish brown. By naturally cooling down to room temperature and drying for 12 h in an oven, a dried gel was formed. Subsequently, the dried gel was reheated to 230 °C for 2 h until it transformed into a black
resinous precursor. Finally, the SmAlO$_3$ powder was obtained after calcining the precursor in air at various temperatures.

The suggested chemical reaction is as follows:

\[
6\text{CH}_3(\text{CH}_2)_{16}\text{COOH} + 2\text{Sm(NO}_3)_3 \rightarrow 2[\text{CH}_3(\text{CH}_2)_{16}\text{COO}]_3\text{Sm} + 3\text{H}_2\text{O} \uparrow + 6\text{NO}_2 \uparrow + 3\text{O} \uparrow
\]

\[
6\text{CH}_3(\text{CH}_2)_{16}\text{COOH} + 2\text{Al(NO}_3)_3 \rightarrow 2[\text{CH}_3(\text{CH}_2)_{16}\text{COO}]_3\text{Al} + 3\text{H}_2\text{O} \uparrow + 6\text{NO}_2 \uparrow + 3\text{O} \uparrow
\]

\[
[\text{CH}_3(\text{CH}_2)_{16}\text{COO}]_3\text{Sm} + [\text{CH}_3(\text{CH}_2)_{16}\text{COO}]_3\text{Al} + 39\text{O}_2 \rightarrow \text{SmAlO}_3 + 15\text{H}_2\text{O} \uparrow + 36\text{CO}_2 \uparrow
\]

2.2 Samples preparation

The as-synthesized SmAlO$_3$ powders along with a small amount of PVA binder were granulated and then pressed cylindrical green samples with a size of about Φ10 mm × 5 mm. Finally, these as-pressed cylinders were pressureless sintered for densification at 1350–1550 °C for 4 h.

2.3 Characterization and measurements

XRD patterns were collected using a powder X-ray diffraction analyzer (Bruker D8, Germany) with CuKα radiation at 2θ = 20–80 °. Thermal analysis of dried gel was employed on a Shimadzu DTG-60H instrument in a temperature range of 30–900 °C in air with a heating rate 10°/min. FT-IR spectra were obtained on a Nicolet 6700 FT-IR device by the KBr pellet method ranging from 400 to 4000 cm$^{-1}$. FE-SEM was used to evaluate the morphology and microstructure by depositing a gold coating on the surface of samples. The particulate properties of synthesized powders were examined using a transmission electron microscopy (JEOL-200). According to the Archimedes method, the bulk densities of sintered ceramics were measured. Relative dielectric constant and dielectric loss of sintered microwave ceramics were evaluated using the Hakki-Coleman DR method by a vector network analyzer (Agilent 8722ET) [20, 21]. TC$\rho$ was determined according to the Eq. (1):

\[
\text{TC} \rho = \frac{f_{80} - f_{20}}{f_{25}(80-20)}
\]
Where \( f_{25} \) and \( f_{80} \) denote the resonant frequencies at 25 and 80 °C, respectively.

3. Results And Discussion
3.1. Thermal decomposition process
In order to understand the synthesis process of SmAlO\(_3\), the thermal analysis including TG-DTA for the dried gel was examined and the results are shown in Fig. 2. The endothermic peak in the DTA curve at 110 °C is due to the volatilization of residual water and the melting of gel, accompanied by a weight loss of about 13.3% between room temperature to 230 °C in the TG curve. When the temperature is over 230 °C, all other peaks are exothermal, which indicates that a series of oxidation and combustion reactions are involved in the thermal decomposition process. In the second step, a huge weight loss (43.9%) at the temperature range of 230–600 °C can be attributed to the combustion of organic substances in the gel, the formation and decomposition of samarium and aluminum complexes. It can be observed from the DTA curve that there are four relative strong exothermal peaks at 318, 388, 465 and 585 °C. Especially, the strongest peak at 585 °C may indicate the decomposition of samarium and aluminum complexes and the formation of some amorphous phases. When the temperature exceeds 585 °C no apparent weight loss is found. However, a weak exothermal peak at 683 °C appears in the DTA curve, indicating that a phase transition from an amorphous phase to a crystalline phase occurs. This means that the minimum crystalline temperature to synthesize SmAlO\(_3\) powders by stearic acid method is approximately 683 °C.

3.2 X-ray diffraction patterns and FT-IR spectra analysis
Figure 3 displays the XRD patterns of SmAlO\(_3\) powders after calcination from 700 to 900 °C for 2 h.

After calcination at 750 °C for 2 h, the product exhibits good crystallinity, which can be characterized by some sharp diffraction peaks. These XRD peaks corresponding to the reflections from the orthorhombic phase are good agreement with the database in JCPDS # 71-1597 for SmAlO\(_3\) with perovskite structure. Further increasing the calcination temperature makes the XRD peaks of the powders stronger and stronger. Besides, no any impurity or other intermediate phase is observed in the investigated calcination temperatures. The XRD patterns of SmAlO\(_3\) in our present work are
similar to those of the nanopowders synthesis by polymeric precursor method [19], sol-gel route using malic acid as complexing agent [22]. However, the synthesis temperature of stearic acid method is about 750 °C, which lowers that of the other wet-chemistry routes (800 °C for polymeric precursor method and 950 °C sol-gel technique route). However, this synthesis temperature for the solid-state reaction to obtain pure phase SmAlO₃ is as high as 1400 °C [5]. These evidences indicate that the stearic acid approach is a speedy and energy-saving route for the synthesis of ultrafine SmAlO₃ powders with high sintering activity.

In addition, the refined lattice parameters, lattice strain and crystallite size for the products derived from different synthesis approaches are calculated using the FULLPROF software package and Debye-Scherer’s formula [23, 24]. Table 1 summarized these calculated results.

| Parameters | SmAlO₃-SS | SmAlO₃-H | SmAlO₃-PP | SmAlO₃-SA |
|------------|-----------|----------|-----------|-----------|
| Lattice Parameters (Å) | a = 5.2912 | a = 5.2888 | 5.2672 | 5.2834 |
| | b = 5.2904 | b = 5.2784 | 5.2779 | 5.2866 |
| | c = 7.4740 | c = 7.4733 | 7.4448 | 7.4707 |
| Cell Volume (Å³) | 209.22 | 208.63 | 206.96 | 208.66 |
| Crystallite size (nm) | - | 45 | 29 (2θ = 33.9 °) |

\[ D = \frac{0.89 \lambda}{\beta \cos \theta} \]

Where D, \( \lambda \), \( \beta \) and \( \theta \) are the average crystallite size, X-ray wavelength, full width at half maximum (FWHM) of the diffraction peak and Bragg's angle, respectively.

It is seen that the cell volume of SmAlO₃ phase obtained by wet-chemistry methods is relatively smaller than that of conventional solid state reaction method, indicating that the powder synthesized by wet-chemistry methods has more defects and thus possesses high reaction activity. The calculated crystallite size is 29 nm for the SmAlO₃ powder calcined at 750 °C while taking the diffraction angle 2θ = 33.9 °.
In order to confirm the structural changes between the precursor and the powders underwent calcination process, FT-IR analysis was done and the results are shown in Fig. 4. Small absorption bands at about 2920 cm\(^{-1}\), 1630 cm\(^{-1}\) and 1067 cm\(^{-1}\) in the precursor after heat treatment at 230 °C reveal that little amount of organic substances still exists. With increasing the temperature to 750 °C, no obvious absorption bands of organic substances are observed, as shown in Fig. 4(b). Meanwhile, the absorption bands in the range of 400–700 cm\(^{-1}\) get stronger, which are attributed to the formation of MO\(_6\) octahedra (M = Sm/Al). In addition, a broad band centered at 3427 cm\(^{-1}\) is assigned to the adsorbed water of these powders.

3.3 Morphology observation

Figure 5 shows the scanning electron image of SmAlO\(_3\) powder by calcined the precursor at 750 °C for 2 h. As displayed in Fig. 5, a large size sheet structure is composed of a number of micro/nano-scale crystallites. To draw a reliable conclusion based on the above result, the transmission electron analysis was carried out and the corresponding TEM image is present in Fig. 6. It is observed that nearly all crystallites are almost irregular in shape due to the mutual growth and overlapped shape of adjacent grains. The possible reason is high reactive activity and good sintering activity of the as-synthesized SmAlO\(_3\) nanopowders. As a result, the particle size estimated from the TEM image is about 70 nm, which is significantly larger than that calculated from the Debye-Scherer’s formula.

3.4 Sintering of SmAlO\(_3\)

Using the powder synthesized by stearic acid method as starting materials to prepare SmAlO\(_3\) ceramics for purpose of application as microwave dielectric materials. The synthesized powders were uniformly mixed with 5 wt% PVA solution, pressed some bulk specimens and were sintered at 1350–1550 °C for 4 h.

Figure 7 illustrated the bulk densities of SmAlO\(_3\) ceramics sintered at different temperatures. At 1350–1425 °C, the bulk density drastically increases. But the low bulk densities indicate that the current SmAlO\(_3\) ceramics are not dense. When the sintering temperature is over 1425 °C, the bulk density gradually increases till 1500 °C and then decreases. The maximum bulk density of 6.92 g·cm
$-3$, which is about 96.5% of the theoretical density, was obtained at 1500 °C for 4 h. Compared with the densification temperature of 1650 °C required by the conventional solid state reaction route, the best sintering temperature of SmAlO$_3$ ceramics prepared using the powders by stearic acid method as raw materials can be reduced about 150 °C. In fact, the theoretical density values all exceed 95% at which the influence of pore can be ignored for the microwave dielectric properties. A recent study suggested that a reasonable calcination temperature is critical for obtaining dense ceramics with good microwave dielectric properties [25]. Low or high calcination temperature is disadvantageous to achieve high density ceramics in sintering process. A systematic investigation is worthy to explore for better optimization of density and microwave dielectric properties.

The SEM image of SmAlO$_3$ ceramics at 1500 °C for 4 h is shown in Fig. 8. A dense, uniform microstructure with the average grain size of about 1.8 µm can be observed, which is advantageous to enhance the microwave dielectric properties. In addition, a layered growth steps morphology can also found according to the SEM image. Similar results have been reported in the ceramic systems of 0.75CaTiO$_3$-0.35SmAlO$_3$ and 0.95(Ca$_{0.88}$Sr$_{0.12}$)TiO$_3$- 0.05(Bi$_{0.5}$Na$_{0.5}$)TiO$_3$ [25, 26]. These phenomena have a close relationship with the grain growth and mass transfer in sintering process.

3.5 Microwave dielectric properties of as-prepared SmAlO$_3$ ceramics

The microwave dielectric properties of SmAlO$_3$ ceramics are demonstrated in Fig. 9. As can be seen, the relationship between the $\varepsilon_r$ value and sintering temperature is in firm agreement with that between the density and sintering temperature. The $\varepsilon_r$ firstly increases to the saturated value at 1500 °C and then decreases slightly in the investigated sintering temperature. Also, the $Q\cdot f$ value exhibits a similar behavior. The dielectric constant and $Q\cdot f$ are strongly affected by the pores in the structure before the ceramics are well sintered. Therefore, the $\varepsilon_r$ and $Q\cdot f$ values are obviously reduced when the sintering temperature is below 1525 °C. However, when the sintering temperature is in the range of 1450–1550 °C, the variations in the $\varepsilon_r$ and $Q\cdot f$ values are relatively smaller, which are attributed to high density and uniform microstructure with reduced porosity. The maximum values of
20.22 and 74110 GHz for the SmAlO₃ ceramics at 1500 °C are obtained, respectively.

Figure 9(b) shows the TCf of SmAlO₃ ceramics obtained from the stearic acid method. No any significant rule can be found for the relationship between the TCf and sintering temperature. At 1350-1550 °C, the TCf varies from – 74.04 ppm/°C to -75.94 ppm/°C, which can be explained by the fact that the deciding factor to adjust TCf is mainly the composition of materials.

The sintering properties and microwave dielectric characteristics of SmAlO₃ ceramics derived from various methods are summarized in Table 2. Although the εᵣ and Q·f values obtained in our present work are slightly low than those achieved by polymeric precursor method, the stearic acid method still exhibits some other advantages such as low cost of raw materials, simple and facile synthesis process and low energy consumption. On the one hand, the stearic acid approach significantly reduces the calcination temperature and sintering temperature of SmAlO₃ compared with the conventional solid-state reaction method. On the other hand, the microwave dielectric properties especially the Q·f value are greatly improved. As a result, the stearic acid method is one of the most facile, energy-saving and promising approaches to prepare SmAlO₃ ceramics with good microwave dielectric properties.

| Method | CT (°C) | ST (°C) | RD. (%) | εᵣ | Q·f (GHz) | τᵣ(ppm/°C) | Ref. |
|--------|---------|---------|---------|-----|-----------|-----------|------|
| SS     | 1400    | 1650    | -       | 20.4 | 65000     | -74       | [5]  |
| PP     | 900     | 1550    | 97.2    | 20.94| 78600     | -71.8     | [19] |
| CP*    | 900     | 1550    | 97      | 20.54| 75380     | -69.2     | [18] |
| SA     | 750     | 1500    | 98.7    | 20.22| 74110     | -74.6     | This work |

CP Citrate Precursor; CT Calcination temperature; ST Sintering temperature; RD. Relative density

4. Conclusions

In the current study, SmAlO₃ powder was synthesized successfully by stearic acid method. The characterization results strongly demonstrated that the stearic acid method is one of the facile, low
cost and energy-saving approaches to synthesize well-crystallized SmAlO$_3$ phase after heat treatment at 750 °C for 2 h. The average crystalline size of SmAlO$_3$ powder synthesized at 750 °C was about 70 nm and in an almost irregular shape. The stearic acid method exhibited many advantages over the other wet-chemistry routes such as low cost of raw materials, simple and facile synthesis process and low energy consumption. The stearic acid approach significantly reduced the calcination temperature and sintering temperature of SmAlO$_3$ compared with the conventional solid-state reaction method.

Also, the microwave dielectric properties especially the Q·f value were greatly improved.

Declarations
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Figures
Figure 1

Flow diagram of SmAlO3 synthesized by stearic acid method
Figure 2

TG and DTA curves of the dried gel obtained by stearic acid method
Figure 3

XRD patterns of SmAlO3 calcined at different temperatures for 2 h
Figure 4

FT-IR spectra of the dried gel and the powders calcined at 750 °C
Figure 5

SEM micrograph of SmAlO3 powder calcined at 750 °C for 2 h
Figure 6

TEM photos of SmAlO3 powder calcined at 750 °C for 2 h
Figure 7

Bulk densities of SmAlO3 ceramics sintered at different temperatures
Figure 8

SEM image of the sintered ceramics at 1500 °C for 4 h
Figure 9

Variation in microwave dielectric properties containing $\varepsilon_r$, $Q\times f$ and $\tau_f$