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Morphology-controlled synthesis of quasi-aligned AlN nanowhiskers by combustion method: Effect of NH_4Cl additive

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Abstract

Uniform quasi-aligned AlN nanowhiskers grown in the reacting Al particles have been successfully prepared in high content by combustion synthesis using NH_4Cl as a morphology-controlled promoting additive. FE-SEM and TEM images show that the nanowhiskers, which are single-crystalline hexagonal wurtzite AlN growing along [0 0 1] direction, have diameters in the range of 80–170 nm and a length of several to several tens of micrometers. The effect of NH_4Cl on the growth of nanowhiskers was discussed. It was found that NH_4Cl not only controlled the products' morphology, but also changed the combustion behavior and nitridation mechanism in the combustion synthesis process.
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1. Introduction

In recent years, research on the controlled synthesis of one-dimensional (1D) inorganic nanomaterials has attracted increasing attention because of their importance in both scientific research and technological applications [1–3]. Aluminum nitride (AlN), an important III–V group wide band-gap semiconductor, has aroused great interest due to its high thermal conductivity, good electrical resistance, low dielectric constant, and low thermal expansion coefficient, matching that of silicon [4]. Recently, 1D AlN nanostructures, such as nanowhiskers [5], nanorods [6] and nanobelts [7] were found to process promising applications in both electronics and photonic devices. To date, 1D AlN nanostructures such as nanofibers [8], nanowhiskers [9], nanowires [10–12], nanotubes [13] and nanobelts [7], have been synthesized by several methods, including chemical vapor deposition (CVD), carbothermal reduction and nitridation (CRN), direct nitridation (DN) and DC arc discharge. For these methods, high temperature, catalysts, substrates or long-term production cycle tend to be required, which will increase the cost and limit the applications. From this point of view, it is imperative to further exploit some synthetic routes for preparation of 1D AlN nanostructures.

In addition to above methods, the combustion synthesis (CS, also known as self-propagating high temperature synthesis or SHS), has become a promising choice for industrial fabrication because of its low processing cost, high energy efficiency and short reaction period. To date, many CS processes have been developed for synthesis of pure AlN powders [14–17]. However, since the high thermal-gradient and fast reaction speed in the CS process, as well as the low melting temperature of metal Al (~660 °C), the morphology of the AlN product was difficult to control and often consists of various grain morphologies such as agglomerated particles, whiskers, faceted particles, rods, pyramids, etc. [18–21]. Although a uniform morphology is very important to engineer the properties of AlN-based materials or devices, a morphology-controlled synthesis condition is still difficult to realize, which prevents the CS process from wide application. It has been reported that the addition of ammonium halides (NH_4X, X = F, Cl, Br and I)

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to the starting Al powder during CS can not only control the process parameters, but also enhance the nitridation rate as well as promote the growth of 1D structures (such as AlN fibers and whiskers) [22–25]. This indicates the possibility to achieve the morphology-controlled CS of AlN by NH_4X promoting additives.

In previous studies [26,27], we have reported the synthesis of AlN nanofibers and nanowhiskers with uniform morphologies by the CS method, where the morphologies of products were controlled through adding NH_4Cl and various oxide additives (such as Y_2O_3 and CaO). The oxide additives play an important role for the growth of 1D AlN nanostructures. However, the performances of AlN products, especially the thermal conductivity, would be decreased since the oxygen impurities introduced into the AlN lattice [28]. Moreover, the content of 1D AlN nanostructures in the product is still low.

To overcome the above-mentioned shortcomings, in present paper, uniform quasi-aligned AlN nanowhiskers were successfully formed inside the reacting Al particles by controlling the CS process just using NH_4Cl as the promoting additive. The effect of NH_4Cl on the combustion reaction process and morphology evolution of the products was investigated. Finally, by repeating the combustion reaction two times using the as-synthesized AlN powders as diluent, high content of the AlN nanowhiskers in the final products was achieved.

2. Experimental procedure

The starting materials were high-purity Al (>99.9%, ~23 μm, Toyo Aluminum K.K., Tokyo, Japan), AlN diluent powder (type H, >99.9%, ~0.5 μm, Tokuyama K.K., Hino, Tokyo, Japan), and NH_4Cl additive (>99%, Nacalai Tesque, Inc., Kyoto, Japan). The morphological characteristics of Al powders and AlN diluent are shown in Fig. 1. In a typical experimental procedure, Al and AlN powders were mixed with a molar ratio of 4:6, which was chosen according to a previous study to achieve looser and full AlN product [29]. Additionally, 6 wt% NH_4Cl was also added as a promoting additive to control the morphology of the product. The powders were lightly mixed using mortar for 10 min, and then sieved through a 212-mesh sieve to disperse any large agglomerates. The mixture (50 g) was poured into a porous graphite container (Ø 42 mm × 90 mm H) at a tapping density of 0.6 g/cm^3. Then, the container was placed into a combustion chamber, and two W-Re thermocouples protected by alumina tubes were inserted into the center of the mixture (one at the middle and the other near the top surface) at a fixed distance of 30 mm to record the temperature–time pattern of the combustion and determine the combustion speed by measuring the time lapsed for the wave passage between the two thermocouples. The chamber was evacuated and then filled with high-purity N_2 (99.99%) at the pressure of 1 MPa. The mixture was ignited from the bottom with an ignition pellet (2 g, Al/AlN = 1/1 wt%) by passing an electric current of 60 A × 20 V for 10 s through a carbon ribbon under the pellet. After the first combustion reaction, the as-synthesized AlN product, which was a loose cake composed of quasi-aligned nanowhiskers and original AlN diluent, was broken lightly by a mortar. Then, the product was sieved through 212-mesh sieve to use as the diluent for the second combustion reaction with similar conditions as the first time. The procedures were repeated for two times. Finally, AlN nanowhiskers with high content in the final product were achieved.

The phase purity of the as-synthesized products was examined by using powder X-ray diffraction (XRD; JDX-3530, JEOL, Tokyo, Japan) with Cu Kα radiation. The morphology of the as-synthesized products was observed by field emission scanning electron microscopy (FE-SEM; ERA-8800, ELIONIX, Tokyo, Japan) equipped with energy-dispersive X-ray (EDX) spectroscopy. Samples for SEM observations and EDX analyses were coated with thin films of sputtered gold to reduce electrical charge-up. Transmission electron microscopy (TEM; JEM-2010, JEOL, Tokyo, Japan) was used for further characterization of the products, where both TEM images and selected area electron diffraction (SAED) patterns were acquired.

3. Results and discussion

Fig. 2 shows a typical XRD pattern of the as-synthesized product. All the diffraction peaks can be indexed to the hexagonal wurtzite structure of AlN crystal (JCPDS No. 25-1133). No characteristic peaks of impurities were detected in the pattern. The sharp diffraction peaks indicated the good crystallinity of the product.

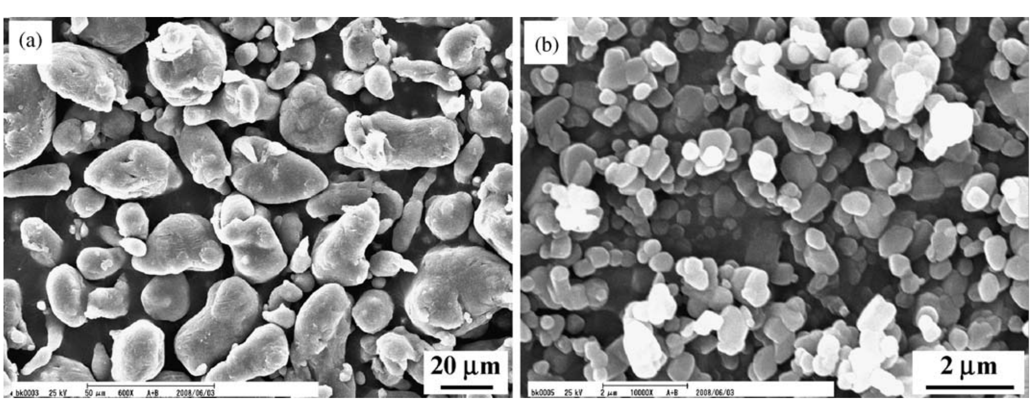

Fig. 1. Morphological characteristics of starting powders: (a) Al and (b) AlN diluent.

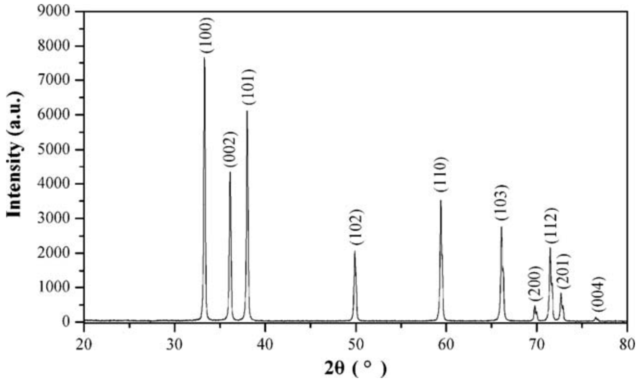

Fig. 2. XRD pattern of the as-synthesized AlN product.

In order to observe the morphology and microstructure of the product by FE-SEM and TEM, the samples were taken from the top surface, side surface and middle center of the product cake. Similar microstructures were observed from these locations. The typical morphologies are shown in Fig. 3. Fig. 3(a) is a low-magnification FE-SEM image which clearly shows that the AlN powders in the product highly disperse in the space without any aggregations, and two major types of morphologies can be clearly seen: one is irregular particles, which are similar size and shape as original AlN diluent (see Fig. 1(b)); the other is ball-like particles which are similar size and shape as original Al particles (see also Fig. 1(a)). Fig. 3(b)–(d) show the detailed FE-SEM images of a single ball-like particle, a cross-sectional view and a broken part of the particle, respectively. It can be observed that the particle is covered with a thin AlN shell (~200 nm), and numerous quasi-aligned AlN nanowhiskers grow from the shell into the interior. The as-synthesized nanowhiskers are shown more clearly in high-magnification FE-SEM image in Fig. 3(e), which shows the AlN nanowhiskers have the diameters in the range of 80–170 nm and a length of several to several tens of micrometers. In FE-SEM observation, there was no grain growth or sintering for both the formed and original AlN diluent particles, and the uniform quasi-aligned AlN nanowhiskers were formed mainly inside the reacting Al particles.

Fig. 3(f) illustrates a representative TEM image of AlN nanowhiskers and corresponding SAED pattern. It indicates that the diameters of AlN nanowhiskers are about 80–170 nm in accordance with the FE-SEM image (Fig. 3(e)) and the nanowhiskers are quite straight with uniform diameters along their lengths. In addition, since no droplets are observed at the tip of the nanowhiskers, the growth of the AlN nanowhiskers was probably governed by a VS (vapour–solid) mechanism. The corresponding SAED pattern (Fig. 3(f), inset) indicates that the AlN nanowhiskers are indeed a single crystal with hexagonal wurtzite structure and grow along the [0 0 1] direction.

It has been proven that NH_4Cl is a versatile, convenient and low-cost promoting additive for CS of 1D AlN micro or nanostructures [24,25], where the NH_4Cl can act as a catalyst, nitrogen source and diluent agent. However, the growth-mode for the quasi-aligned AlN nanowhiskers in present study is obviously different with the observed before [24,25]. Therefore, what is the effect of NH_4Cl for the growth of this kind of unique nanowhiskers inside reactant Al particles should be studied clearly.

To investigate the effect of NH_4Cl on the formation of quasi-aligned nanowhiskers, several parallel experiments were performed. The CS procedure was similar to the typical process described above, except for the different amount of NH_4Cl or none of it. According to the different amount of NH_4Cl additive (such as 0, 3 and 5 wt%), the process can be briefly noted as AN0, AN3 and AN5, respectively. Therefore, the typical process was noted as AN6. From the FE-SEM observation, we found that without NH_4Cl additive, a broken eggshell-like hollow was obtained (Fig. 4(a)), which was similar as other literatures reported for CS of AlN [30,31]. When the amount of NH_4Cl increases up to 3 wt%, an integrated hollow with porous shell was formed (Fig. 4(b)). With the amount of NH_4Cl further increases to 5 wt%, many irregular micro-rods were grown inside a thin shell (Fig. 4(c)), which was a little similar with the ball-like particle shown in Fig. 3(b). Remarkably, all of these structures have similar size or shape as original Al particles, indicating that they are evolved from original Al particles.

These experimental results show that NH_4Cl plays important roles in the controlled synthesis of AlN products with different ball-like morphologies in our experiments. Fig. 5 shows the typical temperature–time histories for the CS process with different amount of NH_4Cl. As can be seen, all of the combustion temperatures are much higher than the melting point of Al particles. Therefore, the Al particles melted, followed by coalescing to a ball-like form due to the surface tension [32]. The nitridation occurs at the surfaces of these molten Al particles forming nitride shells surrounding the molten Al [23]. For the case of the experiment without NH_4Cl additive, once the combustion reaction triggers, the temperature increases rapidly close to its apex. Due to the high thermal stress, the inner molten Al expands much enough to break the new-formed AlN shell and then pours out rapidly as Al vapor to react with nitrogen gas in a direct nitridation pathway. The reaction can be expressed according to the following equation:

$$Al_{(g)} + N_{2(g)} \rightarrow AlN_{(s)} \quad (1)$$

As a result of high supersaturation of the Al vapor, the as-formed *in situ* hole is covered with a shell consisted of many fine, smooth and homogeneous spherical AlN particles [32]. Therefore, the product with a porous broken-hollow morphology is obtained (Fig. 4(a)). However, with adding NH_4Cl into the starting materials and increasing its amount, the combustion process gradually change to a mild way with no explosive mode and have a relatively low heating rate. This change is due to the sublimation and dissociation of NH_4Cl according to the following reaction:

$$NH_4Cl_{(s,g)} \rightarrow HCl_{(g)} + NH_{3(g)} \quad (2)$$

This reaction is endothermic and absorbs sufficient heat from the samples to disturb the direct nitridation of Al particles with N_2

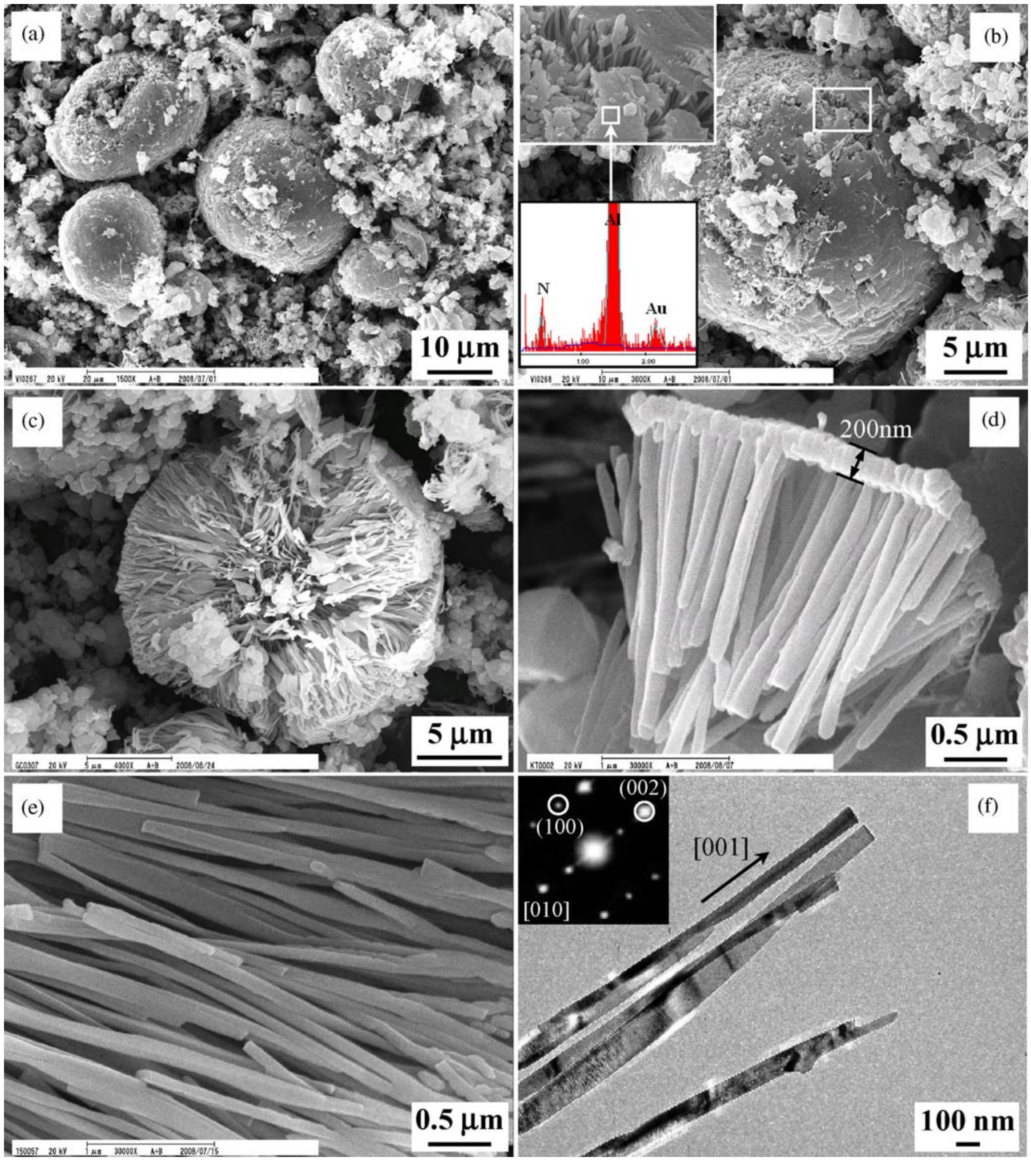

Fig. 3. (a) A low-magnification FE-SEM image of the as-synthesized AlN product with many ball-like particles; (b)–(d) detailed FE-SEM images of a single ball-like particle, a cross-sectional view and a broken part of the particle, respectively; (e) high-magnification FE-SEM image of the as-synthesized AlN nanowhiskers; (f) representative TEM image of the AlN nanowhiskers and corresponding SAED pattern.

gas, which decreases the combustion temperature and retards the wave propagation. Hence, with the amount of NH_4Cl increases, integrated hollow (Fig. 4(b)), ball-like particle with irregular micro-rods (Fig. 4(c)) or quasi-aligned nanowhiskers grown inside (Fig. 3) were obtained in the products of AN3, AN5 and AN6, respectively. The morphological change has strong relationship with the supersaturation of the Al vapor. With the amount of NH_4Cl increases, more and more heat is absorbed. So the combustion temperature decreases, which results in the decrease of supersaturation of the Al vapor. And we found that if

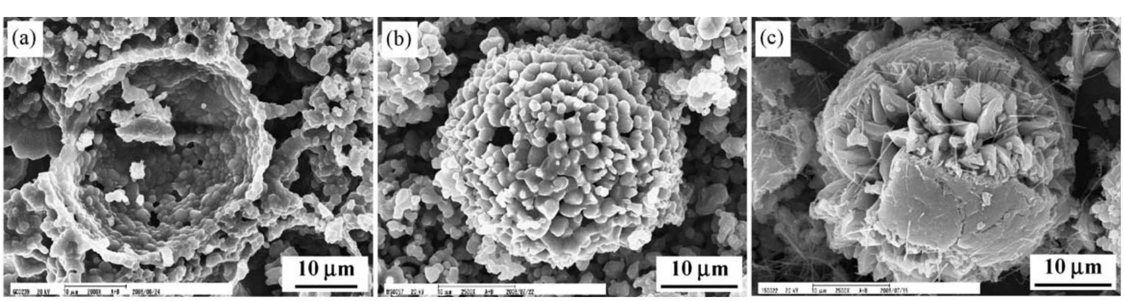

Fig. 4. FE-SEM images of as-synthesized AlN products: (a) AN0; (b) AN3; (c) AN5.

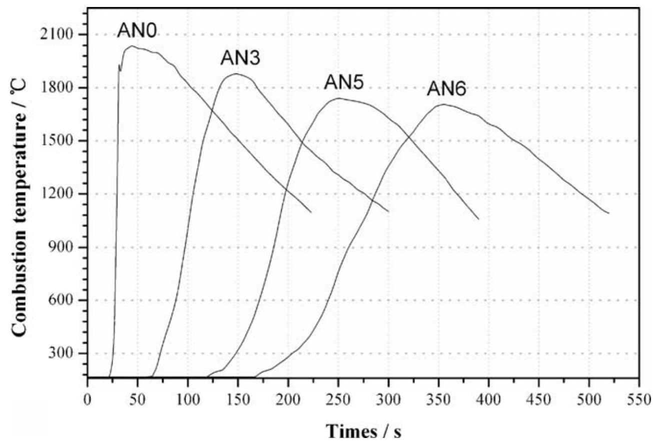

Fig. 5. Typical temperature–time histories for the CS process with different amount of NH_4Cl.

the amount of NH_4Cl in the starting materials higher than 6 wt% in the present work, the combustion cannot be triggered. In other words, it arrived the combustion limit. The combustion limit promotes the formation of ball-like particles with an undeveloped surface as fabricated in the present work, which has been approved by Zakorzhevski and Borovinskaya [16]. Moreover, the addition of NH_4Cl to starting materials offers a different reaction pathway than the direct nitridation mechanism. This different nitridation proceeds via spontaneous chlorination–nitridation sequences similar to the process of direct nitridation of an Al/NH_4Cl mixture as reported by Radwan et al. [33]. The encountered reactions can be described according to the following reactions along with the reaction (2):

$$Al_{(l)} + 3HCl_{(g)} \rightarrow AlCl_{3(g)} + \tfrac{3}{2}H_{2(g)} \quad (3)$$

$$AlCl_{3(g)} + \tfrac{1}{2}N_{2(g)} + \tfrac{3}{2}H_{2(g)} \rightarrow AlN_{(s)} + 3HCl_{(g)} \quad (4)$$

The spontaneous chlorination–nitridation reactions were facile for the growth of 1D AlN nanostructures [11,33]. Therefore, we can understand the growth mechanism of the quasi-aligned AlN nanowhiskers as follows: After the ignition, a thin AlN shell is formed on the surface of Al particle, which can be seen as a micro-reactor and function as a self-catalyze substrate for the growth of nanowhiskers. Then, various gaseous specious such as $HCl_{(g)}$, $NH_{3(g)}$, $N_{2(g)}$ slowly infiltrate through the shell into the molten Al core. The molten Al core is spontaneously halogenated to $AlCl_{3(g)}$ and then nitrided to AlN embryos. Because of the nitridation of Al core is suppressed by the AlN shell, the supersaturation degree is low in the micro-reactor. In this case, the embryos preferentially deposit on nucleated sites and tend to epitaxial growth along [0 0 1] direction by VS mechanism. Therefore, the AlN nanowhiskers are grown with a unique morphology that oriented growth towards the center of the reacting particle. However, because of the relatively higher supersaturation degree [34] (caused by the higher combustion temperature and speed) in the micro-reactor for the sample AN5, many irregular micro-rods are grown inside the reacting particle (Fig. 4(b)).

Based on the above experiments and discussion, the effect of NH_4Cl on the morphology-controlled synthesis of quasi-aligned AlN nanowhiskers by combustion method can be explained, as shown in Fig. 6. First, the combustion behavior can be controlled from an explosive mode to mild by NH_4Cl. With the addition of NH_4Cl from 0 to 6 wt%, both combustion temperature and combustion speed decrease. The combustion temperature decreases from 2038 to 1707 °C, and the corresponding combustion speed decreases drastically from 1.12 to 0.14 mm/s. Second, NH_4Cl gradually controls the conversion of nitridation mechanism from core–shell model by direct nitridation to micro-reactor model by spontaneous chlorination–nitridation pathway. When adding NH_4Cl into the starting materials, both of the two mechanisms affect the nitridation of Al particles. However, with the amount of NH_4Cl increases, the latter mechanism wins the competition. Therefore, controlled CS of uniform quasi-aligned AlN nanowhiskers can be successfully achieved because of the promoting effect of NH_4Cl additive.

However, the content of nanowhiskers in the sample of AN6 is not high enough for the large-scale application (at most

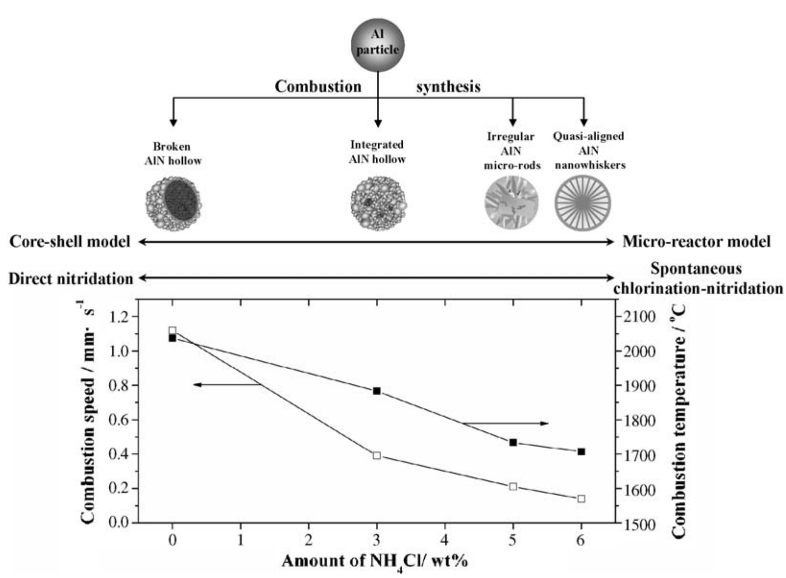

Fig. 6. Schematic illustration of the effect of different amount of NH_4Cl on the combustion behavior, nitridation mechanism and products' morphology.

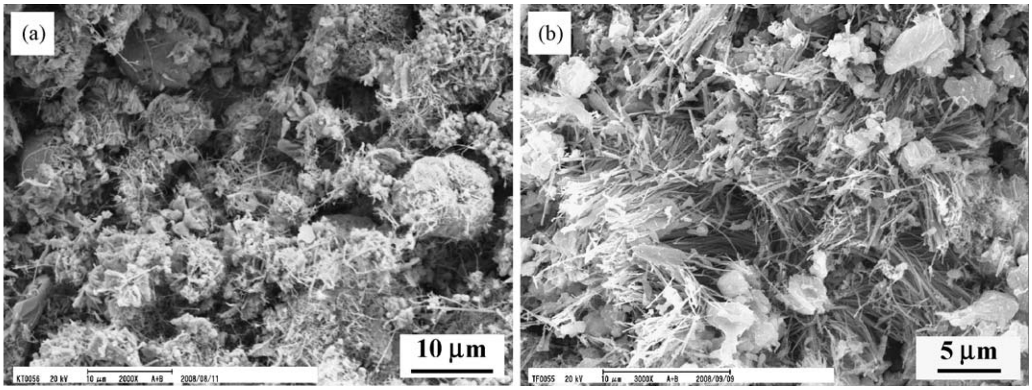

Fig. 7. FE-SEM images of the final AlN product with large-scale nanowhiskers morphology: (a) unground; (b) ground.

40 wt%, same as the mass content of reacting Al powders). In order to increase the content of nanowhiskers in the product, we repeated the combustion reaction two times with similar conditions just using the as-synthesized AlN powders obtained from the former product as diluent. Finally, high content (maximum ~80 wt%) of AlN nanowhiskers in the final product were achieved, as shown in Fig. 7. The rough content of nanowhiskers in the final product was estimated according to the total content of reacting Al powders added into the mixture.

4. Conclusions

In summary, uniform quasi-aligned AlN nanowhiskers with high content have been successfully prepared via controlling the combustion synthesis process with NH_4Cl promoting additive. The as-synthesized nanowhiskers have diameters of 80–170 nm and lengths of several to several tens of micrometers. Based on the experimental results, the effect of NH_4Cl on controlling the combustion behaviors, nitridation mechanisms and morphology evolution of the products are revealed. The present process for synthesis of AlN nanowhiskers is simple, productive, reproducible, energy-saving and can be applied to produce fillers as reinforcement for electrical packaging, structural composites and nanodevices.

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