

# Combustion synthesis method for synthesis of aluminum nitride powder using aluminum containers (II)

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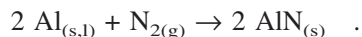
A combustion synthesis method was developed for synthesis of AlN powder. Al powder and small amounts of urea were thoroughly mixed and placed in aluminum containers. A layer of AlN powder was placed on the top of the reactant powders and in between the reactant powders and the container walls. The combustion reaction was ignited by heating the top surface of the powder stack and the aluminum container was converted completely to AlN during combustion reaction. High product yields (~99.5%) were obtained under N<sub>2</sub> pressures of 0.3–0.5 MPa. The product as synthesized is porous and loose and can be easily ground. A quenching technique was developed and formation of the various types of product morphology was investigated. Effects of several process parameters on the product yield were also studied. These results were reported and discussed.

## I. INTRODUCTION

Aluminum nitride possesses many unique properties such as high thermal conductivity, high electrical resistivity, low thermal expansion coefficient, moderately low dielectric constant, good thermal shock resistance, and good corrosion resistance.<sup>1,2</sup> It has been considered for many applications such as substrates for power electronics, an insulating material for radio frequency and microwave packages, an electrostatic chuck dielectric for semiconductor processing, heat sinks for mainframes of super-computers, high thermally conductive composite materials, and hardware for containing or processing molten metals and salts.<sup>3,4</sup> In spite of many desirable properties, AlN has not yet been used widely in industries mainly because of its high market prices due to high production costs. Development of new processes for producing AlN powder at low cost is thus an interesting research topic.

Our research objective has been to investigate the use of a combustion synthesis method self-propagating high-temperature synthesis (SHS) for the production of AlN powder. The SHS method, originally developed in the former USSR,<sup>5</sup> has been applied to synthesis of various materials including ceramics, intermetallics, and composites. Many combustion synthesis processes have

also been developed for synthesis of AlN powder<sup>6–12</sup> and the major reaction can be expressed as



These processes can be divided into two categories according to their methods for holding the reactants. In one category, the reactant powders are pressed into reactant compacts,<sup>6–8</sup> and in the other category, the reactant powders are contained in refractory containers such as graphite crucibles.<sup>9–12</sup>

In our previous study,<sup>13</sup> a new combustion synthesis method has been developed for synthesis of AlN powder. The reactant powders (Al powder + 0.5–1.5 wt% NH<sub>4</sub>Cl) are placed in low-melting-point containers, which are made of thin and perforated aluminum sheets. The aluminum sheet also reacts with N<sub>2</sub> to form AlN during the combustion synthesis reaction. Several problems such as contamination, high nitrogen pressure, and product separation, which are usually encountered in other combustion synthesis methods, are essentially resolved with this method. However, since the product yields (~97%)—and thus the product purities—of this method do not yet meet some application needs, attempts have been made in the present study to improve the product yields.

In this study, our previous process has been improved by placing a layer of AlN powder on the top of the reactant powders and in between the reactant powders and the container walls. The development and the details of the process are described in this paper. Effects of several process parameters on the product yield were investigated and are discussed.

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## II. EXPERIMENTAL

### A. Development of a new process

In our previous method<sup>13</sup> for synthesis of aluminum nitride, the reactant mixtures of Al and  $\text{NH}_4\text{Cl}$  (0.5–1.5 wt%) powders were placed in aluminum containers and the reactant mixtures were heated in  $\text{N}_2$  atmosphere ( $\leq 5$  atm) until ignition of the combustion reaction. The containers were made of perforated aluminum sheet, which had a thickness of  $\sim 0.05$  mm, hole diameters of 0.5–1 mm, and hole areas of 20–30% of the sheet area before punching. The stacking density of the reactant powders was  $\sim 0.35$  g/cm<sup>3</sup>.

Since the decomposition of the added  $\text{NH}_4\text{Cl}$  produces HCl, additional step must be taken to treat the HCl properly to avoid polluting the environment. Another problem of this method is that the outer portion of the combustion product is hard and is difficult to grind due to fusion of the unreacted Al and the product AlN. To resolve these problems, a number of trial experiments were made by employing various techniques. We tried adding small amounts of urea (instead of  $\text{NH}_4\text{Cl}$ ) to the Al powder to avoid the HCl problem. (This thought was brought about by our previous study,<sup>14</sup> in which urea was found to be able to prevent Al particles from coalescing.) The reactant powders were successfully ignited and brought into a complete combustion with the addition of urea. However, the problem with the outer portion of the combustion product remains the same.

In subsequent trial experiments, we found that by placing a layer of AlN powders on the top of the reactant powders and in between the reactant powders and the container walls, the product yield could be significantly improved and the whole product became loose and could be easily ground. A new and better process has thus been developed for synthesis of AlN. All the synthesis experiments reported in this work were thus carried out by using the mixtures of Al and urea powders as reactants and placing a layer of AlN powder on the top of the reactant powders and in between the reactant powders and the container walls.

### B. Description of present experiments

In Table I, the characteristics of the reagents used in the present study are listed. Al and  $\text{CO}(\text{NH}_2)_2$  (0–8 wt%, as percentage of the weight of aluminum and urea

powder) powders were thoroughly mixed at the desired proportions and then poured into an aluminum container. A layer ( $\sim 2$  mm in thickness) of AlN powder was placed on the top of the reactant powders and in between the reactant powders and the container walls. The aluminum container was cylindrical in shape with 80 mm in diameter and 100 mm in height. It was made of perforated aluminum sheet, which had a thickness of  $\sim 0.05$  mm, hole diameters of  $\sim 1$  mm, and hole areas of 20–30% of the sheet area before punching. The stacking density of the reactant powders was  $\sim 0.35$  g/cm<sup>3</sup>.

The reaction chamber used in the present study was shown schematically in our previous study.<sup>15</sup> Figure 1 shows the aluminum container containing the reactant powders and the locations of the thermocouples for temperature measurement. The aluminum container was placed on a perforated graphite plate, which was sitting on a  $\text{N}_2$  gas chamber. A portion of the  $\text{N}_2$  gas could thus flow up through the reactant powders to efficiently supply  $\text{N}_2$  for the synthesis reaction. The reactor was evacuated to  $\sim 650$  Pa by flushing with nitrogen between evacuations. After the evacuations, the reactor was back filled with nitrogen at the desired pressure (0.05–0.7 MPa). The combustion reaction was ignited by heating the top surface of the reactant powders with an electric current ( $\sim 1200$  W) passing through a tungsten heating coil. The heating power was turned off after ignition. A regulated flow of nitrogen was supplied to the gas chamber to maintain a constant pressure inside the reactor.

Variations of the temperatures at the top surface and at the center of the powder stack were measured by using 0.127 mm diameter W 3%Re–W 25%Re thermocouples, which were insulated with 1.2 mm diameter alumina tubes. As shown in Fig. 1, thermocouple T1 was held to form an angle of  $\sim 30^\circ$  with the top surface of the powder stack and was pressed down with a small pressure to assure a good contact with the top surface of the reactant powders. Thermocouple T2 was inserted into the center of the powder stack to measure the temperature variation at the center.

The product was removed from the reaction chamber for analysis after cooling to near the room temperature. Formation of the product was determined by x-ray diffraction (XRD) analysis (SEIFERT, XRD7, Freiberg, Germany). The product as synthesized was crushed to under 150 mesh and was then attrition milled using AlN

TABLE I. Characteristics of the reagents.

Reagent	Particle shape	Particle size	Purity (wt%)	Source
Al	Flake	$1.3 \times 1.0 \times 0.025$ mm	99.93	Transmet Inc.
$\text{CO}(\text{NH}_2)_2$	Irregular	$\sim 150$ mesh	99.5	J.T. Baker
$\text{N}_2$ gas			99.9	Yun-San, ROC
AlN	Irregular	$\sim 60$ mesh	99.9	Produced by the authors' laboratory

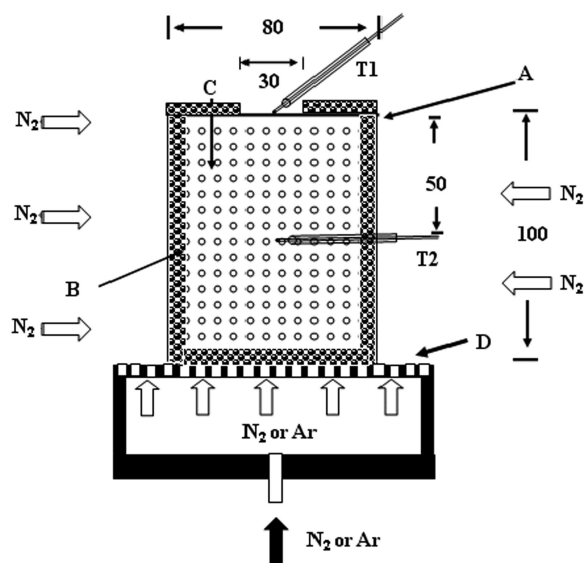


FIG. 1. Schematic illustration of the perforated aluminum container containing the reactant powders and locations of the thermocouples for temperature measurement: (A) perforated aluminum container, (B) AlN powder layer, (C) reactant powders, (D) perforated graphite plate, (T1 and T2) thermocouples. (units are millimeters)

balls (diameter 5 mm) with acetone as grinding fluid. The quantity of residual Al in the product was determined by a gas evolution technique. Approximately 100 ml 15 wt% HCl was added to 5 g combustion product, which had been ground to  $d_{50} \sim 10 \mu\text{m}$ . The quantity of the residual Al was obtained by calculation from the volume of the hydrogen gas thus evolved according to the equation



The product yield was defined as the weight ratio of the Al that had been converted to AlN to that initially contained in the reactant mixtures.

The particle size distribution was measured by using a centrifugal-sedimentation particle size analyzer (Brookhaven, Holtsville, New York, BI-XDC). The morphology of the product was analyzed with a scanning electron microscope (SEM; TOPCON, ABT60, Tokyo, Japan), and elementary analysis was carried out with an energy dispersive x-ray spectrometer. The nitrogen and oxygen contents of the combustion product were determined by using a nitrogen/oxygen analyzer (LECO, Joseph, MI, TC300).

### III. RESULTS

#### A. Combustion phenomena and temperature variations

As the heating power was turned on, evolution of smoke from the outer surface of the powder stack was observed. It began at the top surface of the powder stack

and propagated down during heating and combustion. It was found that ignition (indicated by a glow of red light) took place at the center of the top surface. The combustion wave (referred to as the first combustion) proceeded outward radially, and as it reached the edge of the top surface, it propagated down along the outer surface of the powder stack. Depending on the experimental conditions (e.g., nitrogen pressure and contents of urea), it took 120–200 s for the combustion wave to reach the bottom of the powder stack. During propagation of the combustion wave, a second glow of red light was observed at the center of the top surface. This glow of red light (referred to as the second combustion) soon propagated down and throughout the whole powder stack. This visible radiation of the second combustion was observed to emit mainly from the interior of the powder stack and to last for 140–300 s, depending on the experimental conditions.

Shown in Fig. 2 are typical temperature-time histories measured at the top surface and at the center of the powder stack. The temperature at the top surface (curve a) was seen to increase as the heating power was turned on. After reaching a thermal equilibrium at  $\sim 1100^\circ\text{C}$ , the temperature increased abruptly. This abrupt increase in temperature was coincident with the visual ignition of the first combustion. A second abrupt increase in temperature was seen to occur at  $t \sim 95$  s, which was also coincident with the visual observation of the onset of the second combustion. The second combustion sustained at high temperatures ( $>1500^\circ\text{C}$ ) for  $\sim 40$  s, which is longer than the first ( $\sim 5$  s). The temperature at the center of the powder stack (curve b) did not increase until the onset of the second combustion, indicating that the first combustion did not occur in the interior of the powder stack.

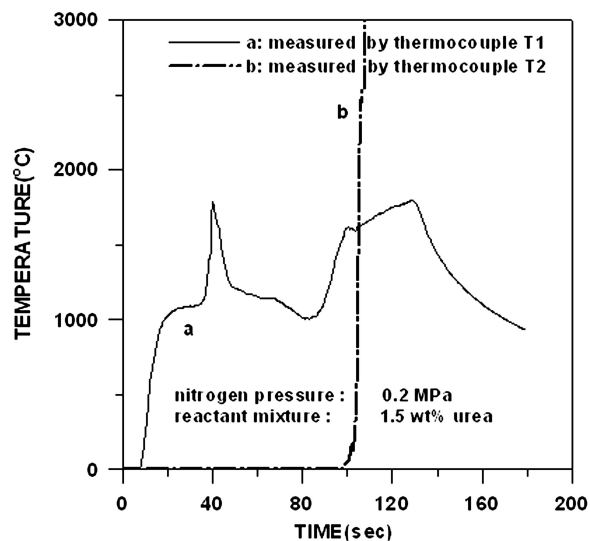


FIG. 2. Typical temperature-time histories measured at the top surface and the center of the powder stack.

After the onset of the second combustion, the temperature at the center of the powder stack increased rapidly and reached so high a value that the thermocouple broke.

## B. Product morphology

After removing the product from the reaction chamber, it was found that the thin, perforated aluminum sheet making up the container had disappeared. The combustion product was highly porous and could be easily ground. Figure 3 is a schematic illustration of the structure of the product as synthesized, and Fig. 4 is composed of eight SEM photographs taken at various locations shown in Fig. 3. The whole product can be classified in four portions, namely the outer layer (A in Fig. 3), the AlN layer (which was added before reaction), the interface layer (B in Fig. 3), and the inner portion (C in Fig. 3 including C-1 and C-2). The outer layer is 0.05–0.1 mm in thickness and white in color, and is composed completely of fibers and whiskers [Fig. 4(a)]. The interface layer is 0.1–0.5 mm in thickness, and pale-yellow in color, and it consists of fiberlike structures [Fig. 4(b)]. The inner portion is yellow in color and can be further classified in two parts, namely the top part (C-1 in Fig. 3) and the central part (C-2 in Fig. 3). Depending on the experimental conditions, the top part has a thickness of 3–6 mm and consists of spherical particles [200–500  $\mu\text{m}$  in diameter, Fig. 4(c)], which are observed to be hollow as shown in Figs. 4(d) and 4(e). The central part consists of twisted flakes with 10–30  $\mu\text{m}$  in thickness [Fig. 4(f)]. All the flakes are composed of granular and needlelike particles [Figs. 4(g) and 4(h)].

Figure 5(a) shows a SEM photograph of the AlN powders which were obtained by grinding the whole product

for 2 h. As can be seen, the particles are irregular in shape and are mostly smaller than 4  $\mu\text{m}$ , which is consistent with the measured particle size distribution shown in Fig. 5(b).

Figure 6 shows typical XRD patterns of the materials taken from various portions of the product as synthesized. As can be seen [Fig. 6(A)], residual Al was detected for the top part. For the interface layer, and the central part of the product, AlN is the only phase detected [Figs. 6(B) and 6(C)]. For the AlN powders obtained from grinding the whole AlN product, the XRD analysis (not shown here) also shows that AlN is the only phase detected.

Table II lists the nitrogen and oxygen contents in different portions of the combustion products. The top part has the lowest nitrogen content ( $30.7 \pm 1.2$  wt%) while the central part has the highest ( $33.1 \pm 1.3$  wt%). The oxygen content is high in the outer layer ( $2.2 \pm 0.08$  wt%) but has a much lower value in the central part ( $0.2 \pm 0.01$  wt%).

To investigate the reaction process, a quenching technique developed in previous studies<sup>16</sup> was used and modified. As shown in Fig. 7, two thermocouples were inserted into the central region of the powder stack. One (T2) measured the temperature at the center (i.e., point A) and the other (T3) measured a point (i.e., point B) 10 mm below the center. In these experiments, as soon as thermocouple T2 broke (due to the second combustion), the  $\text{N}_2$  gas supply was terminated and was replaced by a supply of Ar gas. The reaction chamber was also evacuated simultaneously. The second combustion was soon extinguished due to lack of  $\text{N}_2$  and cooling by Ar. The temperature–time histories at points A and B were measured and shown as curves a and b, respectively, in Fig. 7. As can be seen, the maximum temperature measured at point B was  $\sim 1000$   $^\circ\text{C}$ , which was much lower than point A where combustion had occurred. It is believed that point B was just heated and combustion had not occurred. Point B can thus be considered as in the preheating zone and point A, the combustion zone. The quenched product taken from the combustion zone is yellow-gray in color and consists of twisted flakes [Fig. 8(a)]. All the flakes are composed of agglomerated particles [Fig. 8(b)] and needlelike structures [Fig. 8(c)], among which many spherical particles were also observed [Figs. 8(b) and 8(c)]. With reference to the elementary compositions listed in Table III, the spherical particles are believed to be aluminum particles, which have been partially oxidized and nitrided. Irregular particles [Fig. 8(b)] and needlelike structures [Fig. 8(c)] are believed to be composed of AlN, Al oxides, and unreacted Al. The quenched material taken from the preheating zone is light gray in color and consists of flakes with a porous structure covered on its surface [Figs. 9(a) and 9(b)]. With reference to the elementary compositions (spots G and H

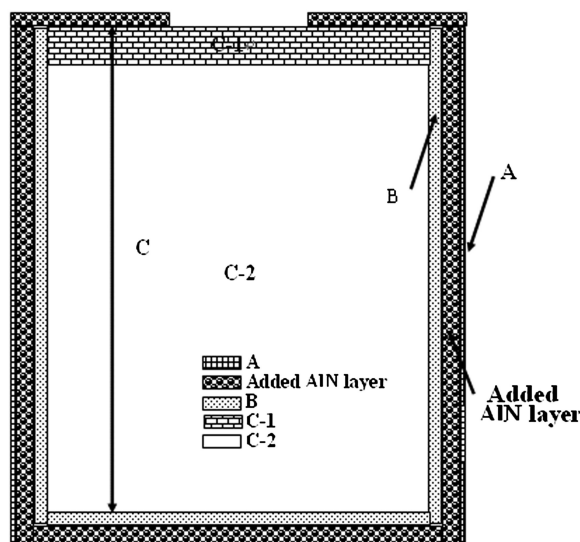


FIG. 3. Schematic illustration of the structure of the product as synthesized: (A) outer layer, (B) interface layer, (C) inner portion, (C-1) top part, (C-2) central part.



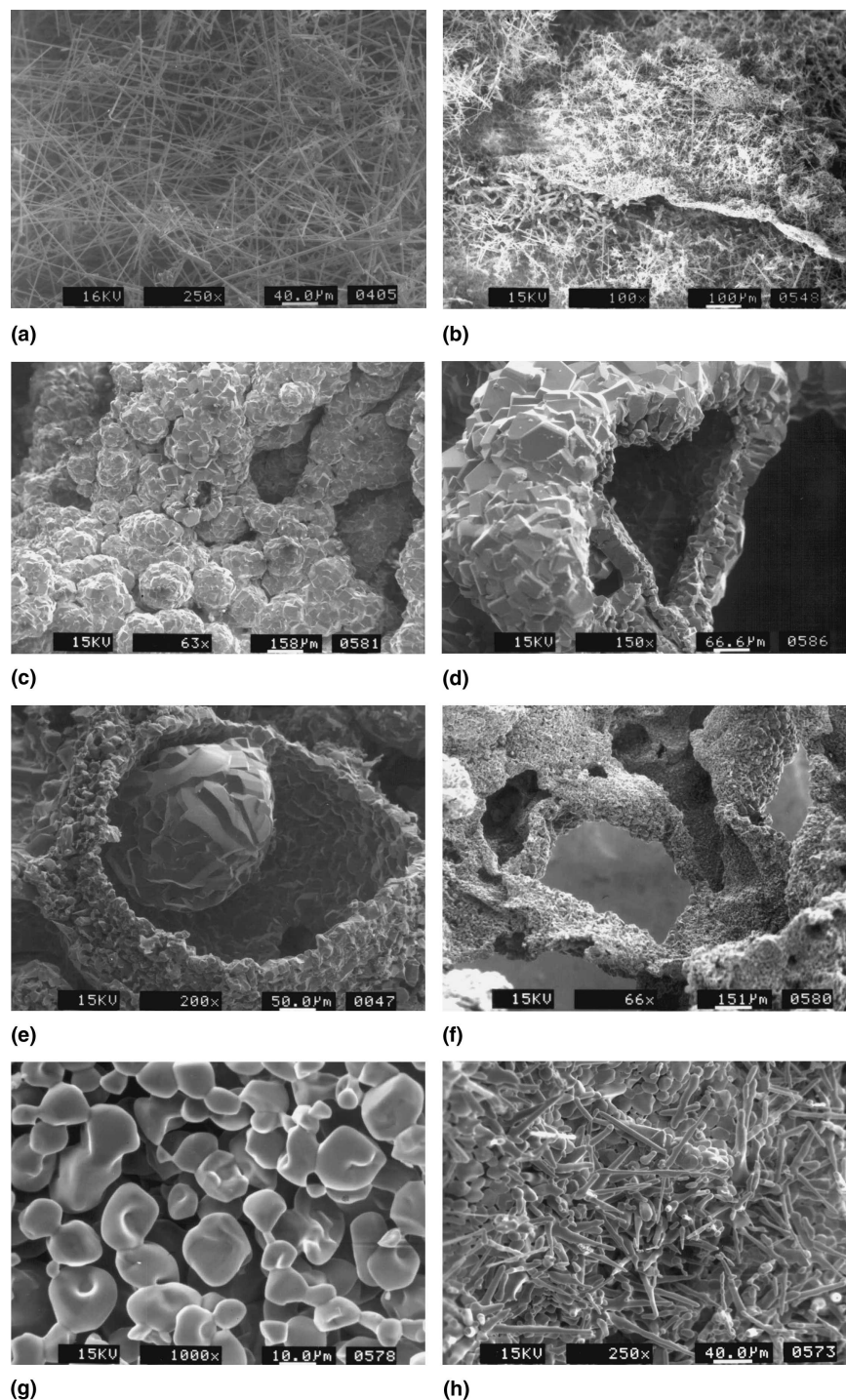


FIG. 4. SEM photographs taken at various locations of the product shown in Fig. 3. (a) outer layer, (b) interface layer, (c–e) top part, and (f–h) central part.

in Table III), the porous structure is composed of Al and its nitrides and oxides.

### C. Effect of process parameters

Figure 10 shows the effect of the urea content on the product yield. As can be seen, high product yields (~99.5%) can be obtained when the urea content is in the

range of 1–2 wt%. The product yield decreases with increasing (>2 wt%) or decreasing (<1 wt%) urea content. In fact, the combustion wave ceases during propagation when the urea content is higher than 6 wt%.

The product yield is seen in Fig. 11 to increase rapidly with increasing  $N_2$  pressure in the range from 0.05 to 0.3 MPa but is relatively unaffected by further increases

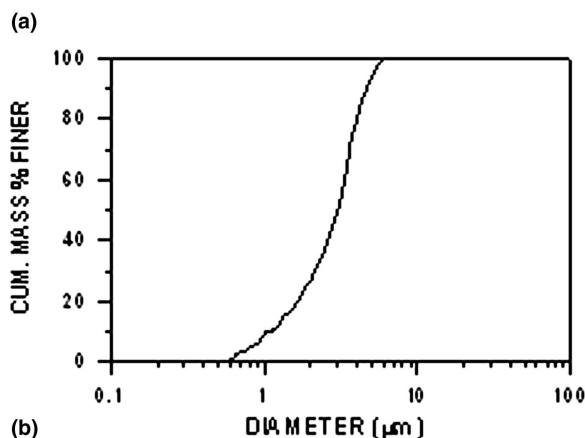
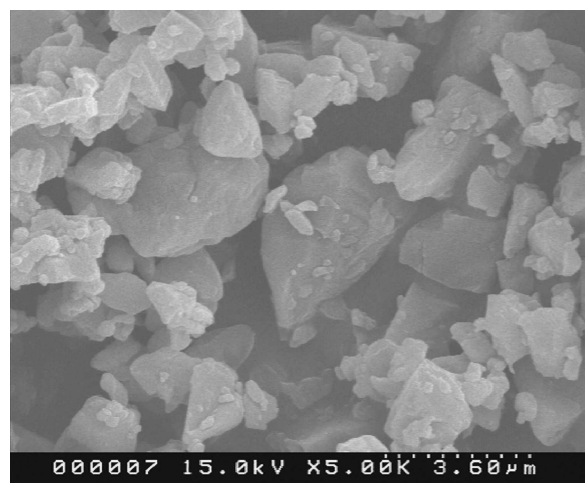


FIG. 5. (a) SEM photograph and (b) particle size distribution of the product after grinding for 2 h.

in nitrogen pressure. The combustion velocity was also observed to increase with increasing  $N_2$  pressure. At nitrogen pressures of 0.1 MPa and less, the combustion wave was observed to propagate in an unsteady manner and, at even lower nitrogen pressures ( $<0.05$  MPa), the combustion could not be ignited.

#### IV. DISCUSSION

##### A. Combustion process and product morphology

As reported in our previous study,<sup>14</sup> the smoke generated during heating was caused by decomposition and sublimation of  $CO(NH_2)_2$ . As mentioned previously, the first combustion was observed to be ignited at the top surface and from Fig. 2, this ignition is seen to occur when the temperature at the top surface reached  $\sim 1100$  °C. The Al particles in a layer of certain thickness at the top of the powder stack must have been heated to above its melting point (660 °C) before combustion. These Al particles are believed to melt and coalesce to form spherical particles. Since the second combustion

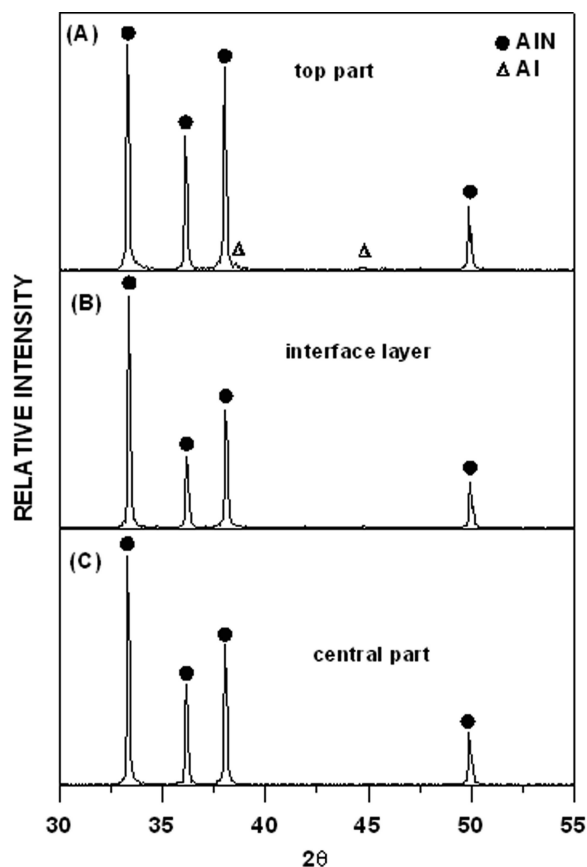


FIG. 6. XRD patterns of the materials taken from various locations in the product as synthesized.

TABLE II. Nitrogen and oxygen contents of the combustion product.

Location	Color	Nitrogen (wt%)	Oxygen (wt%)
Outer layer	White	$30.9 \pm 1.2$	$2.2 \pm 0.08$
Interface layer	Pale-yellow	$32.7 \pm 1.3$	$0.9 \pm 0.03$
Top part	Yellow	$30.7 \pm 1.2$	$0.5 \pm 0.02$
Central part	Yellow	$33.1 \pm 1.3$	$0.2 \pm 0.01$

was also observed to initiate at the top surface of the powder stack, the nitridation of these spherical Al particles was obviously not complete during the first combustion. The nitridation might occur only in a surface layer of the spherical Al particles, leaving residual, molten Al inside a spherical shell of the combustion product. Due to difference in thermal expansion, the AlN shell cracked and the residual, molten Al flowed out to expose itself to  $N_2$ , thus causing the second combustion. This may explain the initiation of the second combustion as well as the formation of hollow spherical structures of the product in the top part [see Figs. 4(c)–4(e)]. Similar combustion phenomena were also observed by Deevi et al.<sup>17</sup> for synthesis of TiN using the combustion synthesis method. Since the Al particles in the top layer

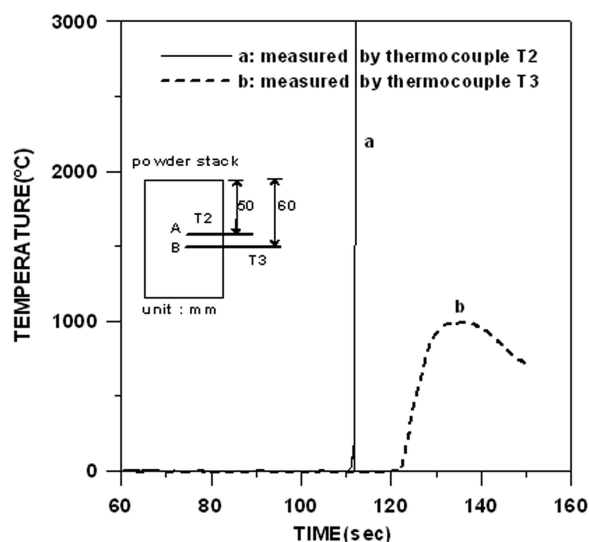
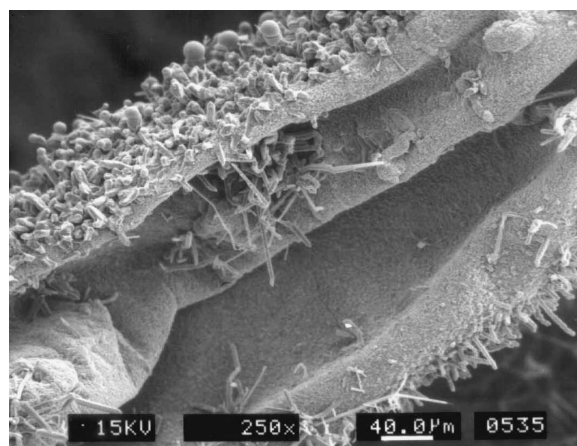


FIG. 7. Typical temperature-time histories measured at the center (marked with A) and at the point (marked with B) ~10 mm below the center of the powder stack when the  $N_2$  gas supply was terminated and replaced by Ar gas.

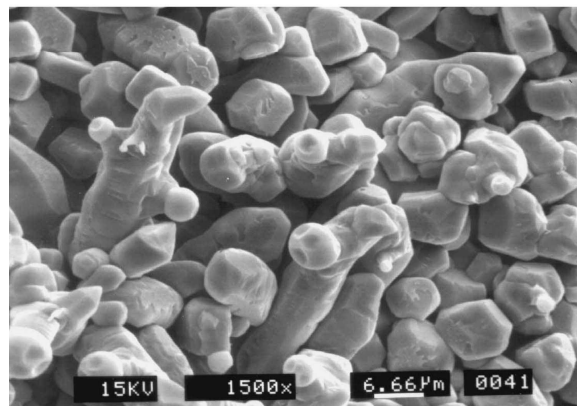
melted and coalesced before combustion, diffusion of  $N_2$  into these Al particles is more difficult due to longer diffusion path. The product in the top part thus had lower nitrogen content (see Table II) and was detected to contain unreacted Al (see Fig. 6).

Perhaps due to cooling effect by the up-flowing  $N_2$  gas stream, the temperature at the center of the powder stack did not increase during the first combustion (see Fig. 2). The first combustion thus occurred only in an outer surface layer but not in the interior of the powder stack. As mentioned previously, the second combustion was also observed to initiate at the top surface and to occur mainly in the interior of the powder stack. The temperature-time histories in Fig. 2 show that the second combustion took a much longer time than the first.

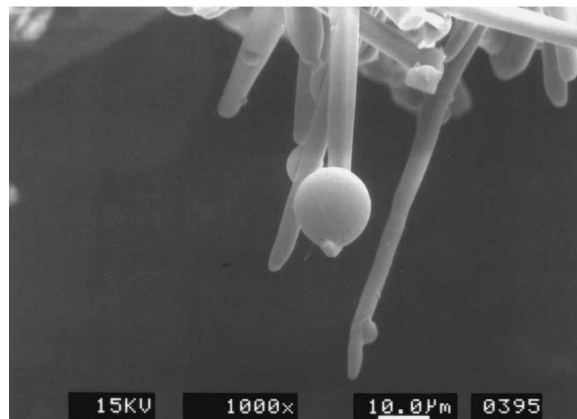
As shown by the SEM photographs of Figs. 4(f) and 8(a), the product of the central part is composed of twisted flakes, a morphology similar to the Al reactant particles. It is believed that the Al particles in the central part did not coalesce during combustion but retained their shapes as flakes (see also Fig. 9). As discussed in our previous study,<sup>14</sup>  $CO(NH_2)_2$  can prevent the Al reactant particles from coalescing by assisting growth of refractory layers on the Al particles. These refractory layers are seen in Fig. 9 to be a porous structure of oxides and nitrides of Al. The molten Al under the refractory layer might flow out through cracks and react with  $N_2$  to form various types of AlN particles [Figs. 4(g) and 4(h)]. The nitrogen required by the combustion reaction could be continuously supplied by flowing of the nitrogen from outside of the powder stack, thus resulting in a complete combustion in the central part.



(a)



(b)



(c)

FIG. 8. SEM photographs of the fractured surface at the reaction zone of the quenched product.

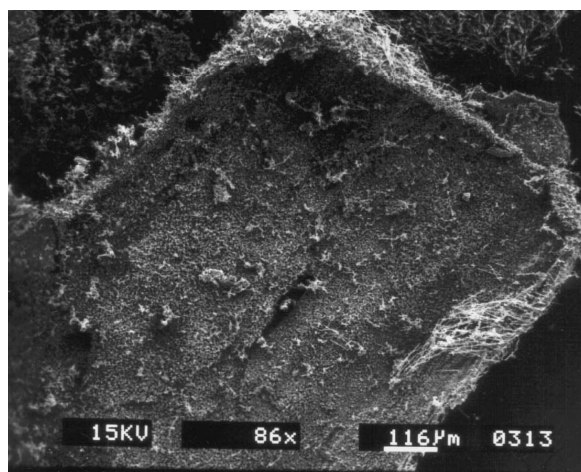
## B. Effects of process parameters

As mentioned previously,<sup>14</sup> urea prevents the Al reactant particles from coalescing by assisting growth of refractory layers on the Al particles. When the urea content is low (<1 wt%), it may not be enough to create refractory layers on all the Al particles. A certain amount of the Al particles may coalesce, thus leading to low product

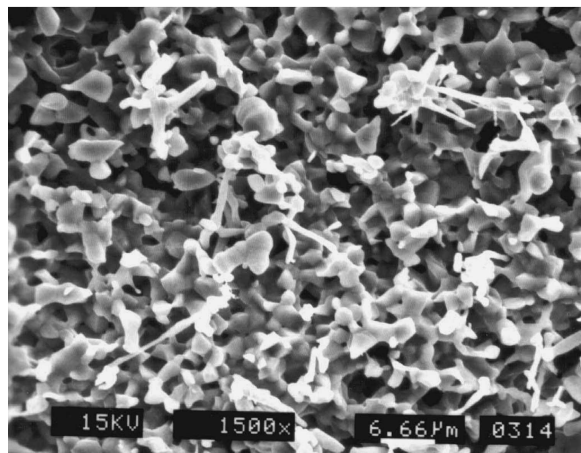


TABLE III. Elementary compositions for the spots shown in Figs. 7 and 8.

Spots in Figs. 7 and 8	Elementary composition (wt%)		
	Al	N	O
A	91.2	6.3	2.5
B	81.5	13.2	5.3
C	87.3	9.2	3.5
D	69.6	27	3.4
E	74.1	24.3	1.6
F	65.7	26.1	8.2
G	54.7	21.8	23.5
H	41.4	25.9	32.7



(a)



(b)

FIG. 9. SEM photographs of the fractured surface at the preheating zone of the quenched product.

yields. When adding high percentages of urea (>2 wt%) to the Al powders, the urea may be more than enough to create refractory layers on all the Al particles. Since decomposition and sublimation of urea is endothermic, the extra urea lowers the combustion temperature, thus leading to low product yields. The optimum content of urea

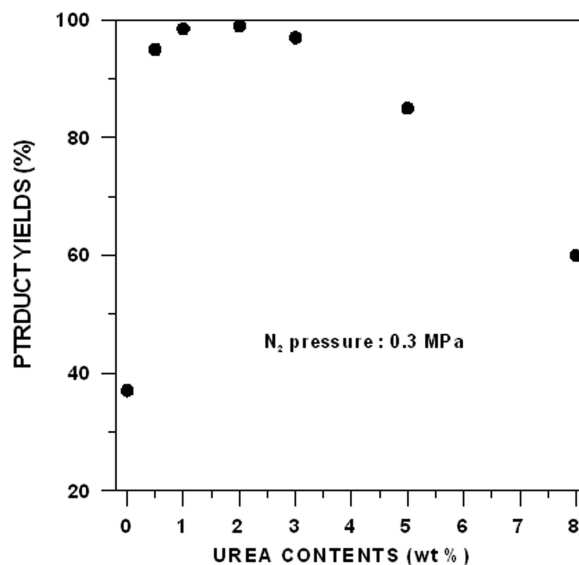


FIG. 10. Effect of urea content on the product yield.

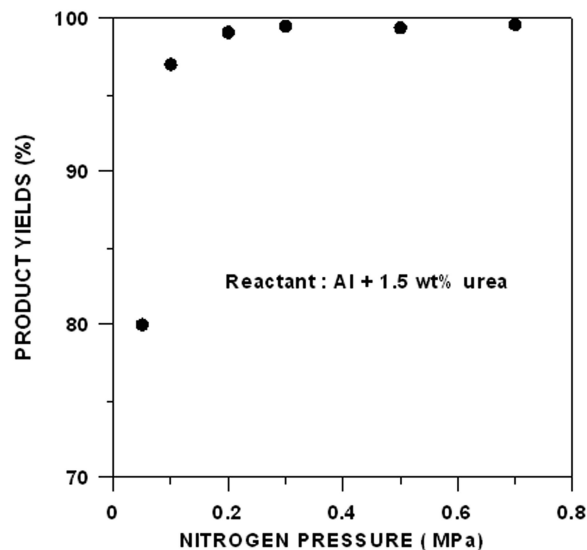


FIG. 11. Effect of nitrogen pressure on the product yield.

was found to be in the range of 1–2 wt% in the present study.

The availability of  $N_2$  inside the powder stack can also be varied by the  $N_2$  pressure. The higher is the  $N_2$  pressure, the more the  $N_2$  is available. The product yield thus increases with increasing  $N_2$  pressure in the low pressure range (<0.3 MPa). The combustion temperature also increases with increasing  $N_2$  pressure in this pressure range because the reaction rate is higher at a higher  $N_2$  pressure. As the  $N_2$  pressure is further increased (>0.3 MPa), the benefit of more availability of  $N_2$  may be compensated by more heat loss to the gas phase. The product yield is thus relatively unaffected as the  $N_2$  pressure is increased to beyond 0.3 MPa.



### C. Advantages of the present process

As reported in our previous study,<sup>13</sup> use of aluminum containers for combustion synthesis of aluminum nitride can minimize or eliminate many problems, which are usually encountered in other combustion synthesis methods.<sup>6–12,14</sup> These include corrosion and environmental problems when using a high content of additives such as  $\text{NH}_4\text{Cl}$ , and contamination and product separation problems when using refractory containers. Compared to the method developed in our previous study,<sup>13</sup> the method developed in the present study presents several additional advantages. First, the corrosion and environmental problems due to use of  $\text{NH}_4\text{Cl}$  (although of a small fraction) in the previous method<sup>13</sup> has been eliminated because it is replaced by  $\text{CO}(\text{NH}_2)_2$ . Second, the product structure has been improved such that the whole of it is porous and loose and can be easily ground. Due to addition of a layer of AlN powder at the top of the reactant powders and in between the reactant powders and the container walls, the thick and hard product layers found in the previous method are eliminated. Third, the product yield has been increased from 97% in the previous method to 99.5% or higher in the present method.

Although addition of the AlN layer at the top of the reactant powders and in between the reactant powders and the container walls creates the above mentioned advantages, the exact functions of the AlN layer are not yet understood. Further studies are being undertaken to investigate the phenomena.

### V. CONCLUSION

An improved combustion synthesis method has been developed for synthesis of AlN powder. The reactant powders (Al + 0–8 wt% urea) are placed in aluminum containers and a layer of AlN powder is placed on the top of the reactant powders and in between the reactant powders and the container walls. The aluminum container also reacts with  $\text{N}_2$  to form AlN during the combustion synthesis reaction. The product is porous and loose and can be easily ground. A product yield of 99.5% or higher can be obtained. Many problems such as corrosion, environment, contamination and product separation, which are usually encountered in other combustion synthesis methods, are essentially resolved by the method developed in the present study. The product yield was found to be affected by the urea content, and the  $\text{N}_2$  pressure.

The optimum conditions to achieve high product yields were found to be 1–2 wt% urea and  $\text{N}_2$  pressure of 0.3–0.5 MPa.

### ACKNOWLEDGMENT

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