A Novel Method of Synthesizing Spherical Nano-structured Composite Particles of Aluminum Nitride using Transferred Type DC Arc Plasma

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Extended Summary

Downsizing of electrical equipment has meant that components of such equipment now require insulating materials with high thermal conductivity. Aluminum nitride (AlN) composite particles consisting of nano-particles dispersed on the surface of spherical micro-particles are one of the materials necessary for developing solid insulating materials with high thermal conductivity. The conventional method for synthesizing the composite particles requires the multi-step process of synthesizing the spherical micro-particles separately from the nano-particles, and then mixing the two kinds of particles. This paper describes a novel simple method of synthesizing AlN composite particles using transferred type DC nitrogen arc plasma. The influence of operating parameters on the shape of the synthesized particles, the diameters of micro-particles and nano-particles in the synthesized particles was clarified.

A transferred type DC arc plasma was generated between a tungsten cathode of a plasma torch and a graphite anode in a chamber. The raw material (crushed angular AlN particles, below 25 µm) was injected into the arc plasma through the cathode using a plasma gas (nitrogen [N2]). By varying the flow rate of the plasma gas and the plasma length, the travelling time of raw particles in the arc plasma was controlled. A suction pipe was positioned downstream from the anode. A reacting/quenching gas (ammonia [NH3]) was blown into the suction pipe.

Figures 1(a) and (b) show SEM photographs of the raw and synthesized particles, respectively. The shape of the raw particle was angular and that of the synthesized particle became relatively spherical. The degree of circularity of synthesized particles increased to approximately 0.9 when the travelling time of raw particles in the arc plasma tP increased beyond 2 ms. The synthesized particle with a circularity of 0.9 was relatively spherical. The AlN content in the synthesized composite particles was 99.7%. TEM observation clarified that many small particles (nano-particles, less than 100 nm) adhered uniformly to the surface of the synthesized particle. It was considered that these nano-particles were synthesized from the vapor formed by raw particles. The circular plots in Fig. 2 show the diameter of micro-particles. The average diameter of the micro-particles in the synthesized particles decreased as the travelling time of raw particles in the arc plasma tP increased. The broken curves in Fig. 2 show the calculated change in particle diameter owing to the evaporation of raw particles in the arc plasma. Calculation and experiment results in Fig. 2 suggested that the raw particle passed thorough the arc plasma where the average temperature was around 3,500 K. As shown in Fig. 3, the diameter of the nano-particles (circular plots) decreased when the flow rate of reacting/quenching gas FQ increased. The broken curve in Fig. 11 shows the calculated change in diameter of nano-particles using a Brownian collision-coalescence model when the diameter for the case of FQ = 5 litres/min was used as the basis. The calculated result showed a similar tendency to the circular plots.
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Aluminum nitride (AlN) composite particles consisting of nano-particles dispersed on the surface of spherical micro-particles are one of the materials necessary for developing solid insulating materials with high thermal conductivity. This paper describes a novel method of synthesizing AlN composite particles using transferred type DC nitrogen arc plasma. The shape of the synthesized particles, the diameter of micro-particles and the volume fraction of micro-particles in the synthesized composite particles were controlled simply by varying the travelling time of the raw AlN angular particles in the arc plasma. The travelling time was controlled by varying the plasma length and the plasma gas flow rate. The diameter of the nano-particles in the synthesized composite particles was controlled simply by varying the travelling time of the raw AlN particles in the arc plasma, and varying the temperature and the vapor density in the nano-particle synthesis space. The temperature and the vapor density were controlled by varying the flow rate of reacting/quenching ammonia gas. The AlN content in the synthesized composite particles was higher than 99%.

Keywords: transferred type arc plasma, aluminum nitride, composite particles, spherical micro-particle, dispersed nano-particle

1. Introduction

Downsizing of electrical equipment has meant that components of such equipment now require insulating materials with high thermal conductivity. To develop polymer insulating materials with high thermal conductivity, it is necessary to add large quantities of ceramic particles with high thermal conductivity, e.g., aluminum nitride (AlN), to organic insulating materials. Mixing small-grained particles with large-grained particles is considered a good way to achieve a high ceramic particle content. The small-grained particles have such a large surface area that they tend to agglomerate with each other. Therefore, it is difficult to achieve a high ceramic particle content when large-grained particles and small-grained particles are mixed and added to organic insulating material. Hence, using composite particles consisting of small-grained particles dispersed on the surface of large-grained particles is considered a good way to achieve a high ceramic particle content. For example, an acrylic resin with a silica particle content of 86%wt was produced by using composite particles consisting of nano-particles dispersed on the surface of spherical micro-particles\(^2\). However, the conventional method for synthesizing the composite particles requires the multi-step process of synthesizing the spherical micro-particles separately from the nano-particles, and then mixing the two kinds of particles\(^2\). Another method uses a spray fluidized bed granulator and requires only one process to synthesize the composite particles, but requires a long process time because the composite particles are synthesized by the gradual agglomeration of small particles\(^3\). Therefore, another simple method for synthesizing the composite particles needs to be developed. Also, in order to synthesize the insulating materials, it is necessary to clarify the optimal composite particle characteristics, that is, the shape of the synthesized particles, the volume fraction of micro-particles in the synthesized particles and the diameter ratio of micro-particle to nano-particle. For example, the viscosity of a epoxy resin mixed with silica particles had a minimum value when the volume fraction of micro-particles in the particles was 60-70%\(^4\). Therefore, it is important to develop a simple method of controlling the characteristics of synthesized composite particles.

There are many methods of synthesizing nano-particles. However, the low production capacity of these methods makes production extremely expensive. The method that uses an arc plasma is a good choice in terms of raising the production rate because it allows plasma power to be increased with ease. There are many reports of the synthesis of AlN nano-particles using an arc plasma\(^5\)-(9). The production rate will increase if the raw materials are injected into the high power arc plasma continuously. The transferred type arc plasma has a long high temperature zone and the advantage of making it easy to increase plasma power, and therefore has the potential to reduce production costs\(^10\). In a previous paper\(^11\), AlN nano-particles were synthesized by making ammonia gas (NH\(_3\)) react with aluminum (Al) vapor that was generated from Al particles injected into a transferred type arc plasma. We may expect to synthesize composite particles consisting of nano-particles dispersed on the surface of spherical micro-particles simply by controlling the evaporation conditions of the raw material in the transferred type arc plasma and the reacting/quenching conditions of the raw material vapor. This paper describes a novel method of synthesizing AlN composite particles using a transferred type DC arc plasma. The influence of operating parameters on the shape of the synthesized particles, the volume fraction of micro-particles in the synthesized particles and
the diameter ratio of micro-particle to nano-particle is clarified\(^{(12)}\).

### 2. Experimental Conditions and Analysis Methods

#### 2.1 Experimental Setup and Conditions

Figure 1 shows the experimental setup. A transferred type DC arc plasma was generated between a tungsten cathode of a plasma torch and a graphite anode in a chamber. The distance between the two electrodes (plasma length) was 25-75 mm. The cathode consisted of a tube-shaped electrode, and the anode a rod-shaped electrode, both water-cooled. The raw material (crushed angular AlN particles) was injected into the arc plasma through the cathode using a plasma gas (nitrogen \([\text{N}_2]\)). The flow rate of the plasma gas was 5-20 litres/min. By varying the flow rate of the plasma gas and the plasma length, the travelling time of raw particles in the arc plasma was controlled. The diameter of the raw AlN particle was below 25 \(\mu\text{m}\), with a feed rate of 2.1 g/min and a feed time of 1-2 minutes. The DC arc current was 140 A and the arc voltage 100-150 V. A suction pipe was positioned downstream from the anode. The distance between the anode and the tip of the suction pipe was 100 mm. A reacting/quenching gas was blown into the suction pipe. \(\text{NH}_3\) was used as the reacting/quenching gas. The flow rate of the reacting/quenching gas was 5-20 litres/min. The surface of the raw AlN particle would evaporate in the arc plasma and \(\text{Al}\) gas would surround the AlN particle, then the \(\text{Al}\) gas would react with and be quenched by the reacting/quenching gas. Thus composite particles would be synthesized consisting of nano-particles dispersed on the surface of spherical micro-particles. The synthesized particles were collected in a tank. The off-gas was exhausted through a suction pipe, a tank and a filter, using a vacuum pump. The tank contained ethanol in order to prevent the oxidation of the synthesized particles. The pressure in the chamber was 20-50 kPa.

#### 2.2 Method of Measuring Particle Velocity in Arc Plasma and Temperature of Arc Plasma

The flight velocity of the AlN particles in the arc plasma was estimated by video observation. Bright lines were observed in the arc plasma owing to the presence of strongly emitting AlN particles. The exposure time of the video observation was 1 ms. The flight velocity of the particle was estimated from the length of the observed bright lines and the exposure time.

Spectroscopic observation of the arc plasma was carried out. The temperature of the arc plasma was measured by comparing the observed intensities of two nitrogen atom spectral lines, i.e. 694.5 nm and 742.3 nm, assuming that the arc plasma was in local thermodynamic equilibrium (LTE). The upper energy levels of these spectral lines are 13.6 eV and 12.0 eV, respectively. The difference between these upper energy levels was so large that the temperature of the arc plasma could be measured accurately.

#### 2.3 Method of Analyzing the Synthesized Particles

The AlN content in the synthesized particle was analyzed by X-ray diffraction (XRD) using the method of reference \((11)\). The shape and diameter of the synthesized particle were determined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). By image processing of the SEM photograph and using the following equations, the degree of circularity \(C\) and projected area equivalent diameter \(D\) of the synthesized particle were derived,

\[
C = 4\pi S / L^2 \quad \text{................................. (1)}
\]

\[
D = 2 \left( S / \pi \right)^{0.5} \quad \text{................................. (2)}
\]

where \(S\) was the projected area of the particle and \(L\) was the circumference of the particle.

The average diameter of the synthesized composite particles containing micro-particles and nano-particles was measured by the Brunauer-Emmett-Teller (BET) method. Using the average diameter of the synthesized particles and the projected area equivalent diameter \(D\), the diameters of the micro-particles and nano-particles were estimated, respectively, assuming that nano-particles adhered in a uniform layer on all surfaces of the micro-particle.

### 3. Results and Discussion

#### 3.1 Particle Velocity in Arc Plasma and Temperature of Arc Plasma

The flight velocity of particles injected into the arc plasma and the temperature of the arc plasma were measured using the method described in section 2.2. The flight velocity was found to be approximately 10 m/s, 20 m/s and 40 m/s when the flow rate of plasma gas was 5 litres/min, 10 litres/min and 20 litres/min, respectively. The travelling time of the particle in the arc plasma \(t_p\) was determined from the flight velocity of the particle and the plasma length. In Ref. \((13)\), the flight velocity of flyash particles in an argon arc plasma was measured by analyzing the progress of images of the Al atomic line (396.1nm) emission owing to evaporation of the flyash particle. The measured velocity increased in proportion to the plasma gas flow rate, as in the experiment of this paper.

The temperature of the arc plasma in the central-axis was measured approximately 7,000 K. The radial temperature distribution was estimated by fitting with the numerical result\(^{(16)}\) of the radial temperature distribution of a nitrogen arc plasma. By using the radial temperature distribution, the average temperature in the cross-section of the arc plasma was determined to be approximately 3,500 K, assuming that the diameter of arc plasma was 20 mm (video image) and the temperature of the plasma edge was 3,000 K because the radiation intensity of a nitrogen arc plasma increases abruptly when the temperature is above 3,000 K\(^{(15)}\).

#### 3.2 Shape and Surface state of Synthesized Particles

Figures 2(a) and (b) show SEM photographs of the raw and synthesized particles, respectively. The shape of the raw particle was angular and that of the synthesized particle became relatively spherical. As shown in Fig. 3, the degree of circularity of raw particles was 0.75 and that of synthesized particles increased to
were observed in the XRD profile. The AlN content in the synthesized particles was estimated at higher than 99%. However, when the travelling time of raw particles in the arc plasma $t_P$ was relatively long (5 ms) and the flow of the reacting/quenching gas was relatively low (5 litres/min), a weak Al peak was observed in an XRD profile of synthesized particles, although the AlN content in the synthesized particles was higher than 99%. The particles accumulating on the surface of the suction pipe tip were also collected. The AlN content in the particles was approximately 66% and the Al content in the particles was approximately 34% when the travelling time $t_P$ was 5 ms. From these results, the following phenomena were considered to occur. The weak Al peak in the XRD profile of the synthesized particles that were collected in the tank was due to the condensation of non-reacted Al gas which was generated from the evaporation of the raw AlN particles in the arc plasma.

Figure 6 shows the calculated chemical equilibrium composition for an NH$_3$/AlN system. The calculation was carried out using the commercial software “HSC Chemistry”$^{16}$, based on the minimizing the Gibbs free energy of the system. Calculation conditions (molar rate, pressure) were similar to the experimental conditions described in section 2.1. It was assumed that NH$_3$ gas was rich in the suction pipe because only NH$_3$ gas was used as the reacting/quenching gas, although N$_2$ gas was used as the plasma gas in the experiment. The AlN evaporated and decomposed to Al gas and N$_2$ gas when the AlN raw particles were in the arc plasma, where the average temperature was approximately 3,500 K. The
Al gas was converted into AlN solid at around 2,100 K. Figure 7 shows the calculated condensation temperature of non-reacted Al gas for the NH₃/AlN system. The calculation was carried out using the same means as reference (11), assuming that AlN solid was not synthesized. As the non-reacted rate of Al gas increased and the ratio of NH₃/AlN decreased, the condensation temperature of the Al gas increased. When the flow of the reacting/quenching gas was relatively low (5 litres/min) and the travelling time of raw particles was relatively long (5 ms), the ratio of NH₃/AlN would be low and the quantity of Al gas was so great that the rate of non-reacted Al gas would be high, therefore, the condensation temperature of the Al gas would be 1,600-1,800 K. The temperature of the space where the reacting/quenching gas was blown in the suction pipe was measured in reference (11) and found to be 1,600-1,700 K. Hence, the non-reacted Al gas would condense and some Al nano-particles would be synthesized when the travelling time of raw particles was 5 ms and the flow of the reacting/quenching gas was 5 litres/min.

### 3.4 Diameter of Micro-particles

The circular plots in Fig. 8 show the diameter of micro-particles estimated by using the method described in section 2.3. The average diameter of raw particles was 8.6 µm and that of the micro-particles in the synthesized particles decreased as the travelling time of raw particles in the arc plasma increased. The average diameter was 3.2 µm when the travelling time was 5 ms. It was considered that the surface of the raw particles evaporated in the arc plasma and the diameter of the particle therefore decreased as time passed. However, varying the flow rate of reacting/quenching gas from 5 litres/min to 20 litres/min did not greatly influence the micro-particle diameter.

The broken curves in Fig. 8 show the calculated change in the diameter of raw particles owing to the evaporation of raw particles in the arc plasma. The calculations took into account the decrease in the particle diameter owing to the evaporation of raw particles in the arc plasma, using the same method as Ref. (17)-(19). The heat transfer from the arc plasma to the particle was calculated by the same means as Ref. (20) using the Ranz-Marshall equation. The calculation method was summarized as follows. The heat flux q from an arc plasma to a particle was calculated using a heat transfer coefficient h. The h was calculated using the Nusselt number Nu. The Nu was calculated as follows:

\[
Nu = 2 + 0.6 \frac{Re^{1/2} Pr^{1/3}}{Pr^{1/3}}
\]  

(3)

where \(Re\) was the Reynolds number and \(Pr\) was the Prandtl number.

The arc plasma has a radial temperature distribution. The temperature where the raw particles passed through was not obvious. Therefore, in order to simplify the calculation, the temperature of the arc plasma was assumed to be near the 3,500 K determined to be the average temperature of the arc plasma in section 3.1. Calculation and experimental results in figure 8 suggested that the raw particle passed through the arc plasma where the average temperature of the arc plasma was around 3,500 K. Video observation showed that the arc plasma had a tendency to bend when the travelling time of raw particles was 5 ms because the flow rate of plasma gas was relatively low (5 litres/min) and the plasma length was relatively long (50 mm). Therefore, it is considered that the raw particles had a tendency to pass through the relatively low temperature zone of the arc plasma when the travelling time was 5 ms.

### 3.5 Volume Fraction of Micro-particles in the Synthesized Particles

The synthesized composite particles consisted of both small-grained and large-grained particles. Ref. (4) mentions that it is important to control the volume fraction of large-grained particles in the composite particles in order to control the viscosity when mixing the composite particles and the organic insulating materials. This section therefore deals with the volume fraction of micro-particles in the synthesized particles. The volume of vapor from which the micro-particles were produced was calculated using the diameter difference between the raw particles and the micro-particles in the synthesized...
particles. It was considered that all the nano-particles synthesized from the vapor were not collected in the suction pipe of the chamber because the vapor had a tendency to spread in all directions. The collection rate of the nano-particles in the suction pipe to the raw particles injected into the arc plasma was approximately 10% in the previous report\(^{(22)}\). The adherence rate of nano-particles on the micro-particle to all synthesized nano-particles was therefore set at 10% and the volume fraction of micro-particle in the synthesized particles was determined.

The circular plots in Fig. 9 show the volume fraction of micro-particles in the synthesized particles obtained from experiments. The volume fraction of micro-particles decreased as the travelling time of raw particles in the arc plasma \(t_P\) increased. The volume fraction of micro-particles was changed from 80% to 50% by varying the \(t_P\) from 2 ms to 5 ms. The broken curves in Fig. 9 show the calculated results of change in the volume fraction of micro-particles. The calculated result was obtained using the calculated results of figure 8. The temperature of the arc plasma was assumed to be near the 3,500 K measured as an average temperature of the arc plasma in section 3.1. Calculation and experimental results in Fig. 9 also suggested that the raw particles passed through the arc plasma where the temperature was around 3,500 K.

### 3.6 Diameter of Nano-particles

As shown in figure 10, the diameter of the nano-particles in the synthesized particles (circular plots) increased from 20 nm to 40 nm when the travelling time of raw particles in the arc plasma \(t_P\) increased from 1 ms to 5 ms. The diameter \(d\) of the nano-particles was investigated numerically using a Brownian collision-coalescence model as follows\(^{(23)}\),

\[
    d = k \times T_n^{0.2} \times V^{0.4}
\]

where \(T_n\) is the temperature of the nano-particle synthesis space, \(V\) is the vapor density in the nano-particle synthesis space and \(k\) is a constant.

The broken curve in Fig. 10 shows the calculated change in the diameter of nano-particles using Eq. (5) when the diameter for the case of \(t_P = 0.6\) ms was used as the basis. The temperature \(T_n\) was measured in the previous paper\(^{(11)}\). The vapor density \(V\) was calculated using the diameter difference between the raw particle and the micro-particle of the synthesized particle. The calculated result showed a similar tendency to the circular plots in Fig. 10.

As shown in Fig. 11, the diameter of the nano-particles (circular plots) decreased from 80 nm to 40 nm when the travelling time of raw particle \(t_P\) increased from 5 litres/min to 20 litres/min. The broken curve in Fig. 11 shows the calculated change in diameter of nano-particles using Eq. (5) when the diameter for the case of \(F_{\text{RQ}} = 5\) litres/min was used as the basis. The vapor density \(V\) was calculated assuming that \(V\) decreased as \(F_{\text{RQ}}\) increased. The calculated result showed a similar tendency to the circular plots in Fig. 11.
From the results of this section and sections 3.2, 3.3 and 3.4, the mechanism of the synthesis of the composite particles consisting of nano-particles dispersed on the surface of spherical micro-particles was considered to be as follows. When the angular raw particles were injected into the arc plasma, the surface of the raw particles evaporated and their corners were rounded off, making the particles (micro-particles) relatively spherical. The AlN vapor generated in this process decomposed to Al and N, and the Al gas progressed downstream with the AlN micro-particles and reacted with and was quenched by the NH3 gas in the suction pipe, and so that finally AlN nano-particles were synthesized and adhered to the surface of the spherical micro-particles. Due to the mechanism mentioned above, as the travelling time of the raw particles in the arc plasma increased, the degree of circularity of the synthesized particle increased and the diameter of the micro-particle and the volume fraction of micro-particle in the synthesized particle decreased. And the diameter of the nano-particle in the synthesized particle decreased, as the temperature and the vapor density in the nano-particle synthesis space decreased.

3.7 Diameter Ratio of Micro-particle to Nano-particle

As described in section 3.4, the diameter of the micro-particle in the synthesized particles was changed by varying the travelling time of raw particle in the arc plasma \( t_P \). As described in sections 3.4 and 3.6, the diameter of the nano-particle in the synthesized particles was changed by varying the \( t_P \) and the flow rate of the reacting/quenching gas \( F_{RQ} \). As shown in Fig. 12, the diameter ratio of the micro-particle to the nano-particle was changed from 80 to 250 by the flow rate of the reacting/quenching gas \( F_{RQ} \). As shown in Fig. 13, the diameter of the micro-particle to the nano-particle was changed from 0.75 (raw particle) to approximately 0.9 and the shape of the synthesized particle was nearly spherical. The AlN content in the synthesized particles was higher than 99%. TEM observation clarified that many small particles of less than 100 nm adhered uniformly to the surface of the synthesized particle. The volume fraction of micro-particles in the synthesized composite particles were controlled from 80% to 50% by varying the travelling time of the raw particles from 2 ms to 5 ms. The diameters of micro-particles and nano-particles were controllable over a wide range by varying the travelling time of the raw particles from 2 ms to 5 ms and the flow rate of reacting/quenching NH3 gas from 5 litres/min to 20 litres/min. The mechanism of the synthesis of the composite particles was considered to be as follows. When the angular raw particles were injected into the arc plasma, the surface of the raw particles evaporated and their corners were rounded off, making the particles (micro-particles) relatively spherical. The AlN vapor generated in this process decomposed to Al and N, and the Al gas progressed downstream with the AlN micro-particles and reacted with and was quenched by the NH3 gas in the suction pipe, and so that finally AlN nano-particles were synthesized and adhered to the surface of the spherical micro-particles. The experimental results of the particle diameters showed a similar tendency to the calculated results. These results show how to control the characteristics of AlN composite particles by controlling the conditions of their formation.

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References

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