In-flight Al₂O₃ spheroidization process with a constant small power DC-RF hybrid Ar/He plasma flow system

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Abstract: The purpose of this paper is to clarify experimentally the effect of helium gas addition into argon gas on the in-flight alumina spheroidization process by DC-RF hybrid plasma flow system under a small input power. Spheroidization rate is evaluated through SEM image analysis with correlation to added helium gas flow rate, plasma enthalpy, and also particle velocity and particle temperature. Spheroidization rate increases through enhanced heat exchange between Ar/He plasma and particles, and also through decrease of DC jet velocity. Spheroidization rate of 82.7% is obtained with as small as 4 % of helium addition into argon central gas.

Keywords: DC-RF hybrid plasma flow; in-flight particle process; spheroidization; mixing gas

1. Introduction

Direct current (DC) plasma jet and radio frequency inductively coupled plasma (RF-ICP) flow are typically utilized for in-flight particle processes. DC plasma jet has some advantages, such as easy ignition, stability, and simple flow structure. However, there are some disadvantages for particle processing, such as small reaction region, high velocity which leads to less residence time, and so on. On the other hand, RF-ICP flow has some advantages too, which are clean, large volume, and long residence time of in-flight particle. RF-ICP flow also has some disadvantages such as plasma instability and strong back flow resulting from Lorenz force in an RF coil region [1, 2].

Therefore, a DC-RF hybrid plasma flow system, which is the RF-ICP flow assisted by DC plasma jet, is expected to overcome such disadvantages of the conventional plasma flow generation methods [3-5].

In the previous study, only an argon gas is used as a working gas because of easy plasma ignition. The helium gas has advantages in plasma enthalpy and the thermal conductivity, compared with the case of pure argon. Therefore, using Ar/He mixture gas can be expected to enhance the performance of DC-RF hybrid plasma particle processing under a small input power.

The goal of this research is to clarify effect of helium gas addition on the in-flight alumina spheroidization process utilizing a DC-RF hybrid plasma flow system under a small input power. This research is aiming to achieve further high spheroidization rate through effective heat exchange by adding helium gas into argon gas and also by decreasing DC plasma jet velocity. The morphology and size of the in-flight processed powder of Al₂O₃ are characterized through scanning emission microscopy (SEM) observation relating to helium gas flow rate, and also particle velocity and temperature, and plasma enthalpy.

2. Experimental analysis

2.1 Experimental setup and measurement system

Figure 1 (a) and (b) show a schematic illustration of a DC-RF hybrid plasma flow system and a detailed schematic illustration of DC-RF hybrid plasma torch, respectively. The main working gas is argon. DC power is 1 kW (25 V, 40 A) and RF power is 6.6 kW (4 MHz, 6 kV, 1.1 A). The flow rate of swirling sheath gas (Q_sw), central gas for DC plasma jet (Q_c), and carrier gas for powder feeding (Q_carrier) are 20 l/min, 4.2 l/min, and 0.8 l/min, respectively. Helium gas is added into argon gas for either central gas or swirling sheath gas as shown in table 1. Experiments were carried out for different DC torch diameters (d_DC) of 3 mm and 4 mm to clarify the effect of DC jet velocity.

Table 1. Operating conditions of gas flow rate.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Q_c (Ar)</th>
<th>Q_c (He)</th>
<th>Q_sw (Ar)</th>
<th>Q_sw (He)</th>
<th>Q_carrier</th>
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<td>20.0</td>
<td></td>
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<tr>
<td>B</td>
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<td>1.0</td>
<td>20.0</td>
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<td>C</td>
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<td>1.5</td>
<td>20.0</td>
<td></td>
<td></td>
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<tr>
<td>D</td>
<td>2.2</td>
<td>2.0</td>
<td>20.0</td>
<td></td>
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<tr>
<td>E</td>
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<td>19.0</td>
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<tr>
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<td>1.5</td>
<td></td>
</tr>
<tr>
<td>G</td>
<td>4.2</td>
<td></td>
<td>18.0</td>
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</table>
Commercial alumina powder manufactured by Nihon Kenna Kogyo, is injected into DC plasma torch by carrier gas. For in-flight processing, precursor powder is alumina powder with average size of 3.8 μm. For dispersed in-flight particle velocity and temperature measurement, particle tracking velocimetry (PTV) and two-wave length pyrometry at 700 nm and 800 nm are used. Alumina with the average size of 15 μm is injected. The melting temperature of alumina (T_m) is 2,319 K.

### 2.2 Experimental method

The specific enthalpy of the plasma is determined through an enthalpy probe measurement. Plasma enthalpy is evaluated from the difference between the inlet and outlet temperatures of cooling water in the probe according to the following relation;

\[
h_p = \frac{\dot{m}_c C_{pw} \Delta T_c}{\dot{m}_g}
\]

where \( \dot{m}_c \) is mass flow rate of water, \( C_{pw} \) is specific heat of water and \( \Delta T_c \) is temperature difference of coolant at the inlet and the outlet of the probe [6].

In particle spheroidization process, irregularly shaped particles are melted in the thermal plasma and then the molten particles form spherical droplets by surface tension in the liquid phase. The processed spherical droplets are cooled down and form spherical particles after solidification. The spheroidized particles are collected on the particle collector at the bottom of chamber as shown in Fig.1 (a). Spheroidization rate of collected particles is analyzed through SEM images. Spheroidization rate is defined as the number of spherical particles to total particles in the SEM image.
3. Results and discussion

3.1 The effects of helium addition

Figure 2 shows the maximum operating pressure for stable plasma as a function of helium content. DC-RF hybrid plasma flow system can be operated below this maximum pressure. Under the constant input power as described in 2.1, maximum operating pressure decreases through helium addition into either central or swirling sheath gas. Due to the increase in the effective ionization energy by adding helium, an operating pressure is decreased to generate the stable plasma at constant electric power. Helium addition to central gas, the maximum operating pressure is higher than the case of adding to swirling sheath gas. From this result, operating pressure for spheroidization process is determined to be 20 kPa in order to maintain the stable plasma flame under all helium injecting conditions.

Figure 3 (a)-(c) show photographs of DC-RF hybrid plasma flow. DC jet pinches by helium addition into central gas as shown in Fig.3 (b) because of the pinch effect. Figure 3 (c) shows that the bright zone of RF plasma actually shrinks compared with Fig.3 (a).

Figure 4 shows axial distribution of plasma enthalpy. Plasma enthalpy in Ar/He mixture gas is higher than that in a pure argon gas for all the cases. Plasma enthalpy drastically increases especially in downstream of the induction coil at $z = 90 \sim 120$ mm.

Figure 5 shows the temperature and velocity of in-flight particles under pure argon, helium content of 4% added into central or swirling sheath gas, respectively. Helium addition brings about increase of particle temperature because thermal conductivity of helium is higher than that of pure argon.

Figure 6 shows alumina spheroidization rate of in-flight powder processing of Al$_2$O$_3$. Initial precursors with various irregular polygons were spheroidized after in-flight process. Spheroidization rate effectively improves by helium gas addition. Especially, only 6% of helium addition to swirling sheath gas results in improvement of spheroidization rate from 33% to 69% compared with pure argon condition. Thermal conductivity of helium is higher than argon due to the mass difference. In addition, the collision integrals of argon species are higher than helium species [7].

3.2 The effects of reduced particle velocity

Figure 7 (a) and (b) show DC plasma jet with diameter of 3 and 4mm, respectively. Decrease in particle velocity elevates particle residence time and heating time through the expansion of diameter. Figure 7 (b) shows that DC plasma jet widens and elongates compared with Fig.7 (a).
Figure 8 shows particle in-flight velocity and temperature. The mean velocity of in-flight particles decreases from 50.4 to 40.3 m/s by expanding the nozzle diameter of DC torch from 3 mm to 4 mm. The lower particle temperature for 4 mm nozzle may come from the larger particle loading. In general, the particle temperature must increase for 4 mm nozzle under the constant particle loading. Further experiment needs to be carried out.

Figure 9 shows the spheroidization rate for 3 mm and 4 mm nozzle. Larger nozzle diameter reduces flow velocity and then decreasing particle velocity influences spheroidization rate due to longer residence time and heat flux exchange time of in-flight particle in the plasma core. Effect of enlarging nozzle diameter is clearly found from Fig.10. Through enlargement of nozzle diameter, spheroidization rate improves for any helium content. Especially, spheroidization rate of 82.7% is optimized as small as 4 % of helium addition added into central gas. In other words, only enlargement of 1 mm of DC torch nozzle diameter and an even small amount of helium addition drastically improves efficiency of spheroidization process in DC-RF hybrid plasma flow system.

4. Conclusion
The effect of helium gas addition into argon gas on the in-flight alumina spheroidization process utilizing a small power DC-RF hybrid plasma flow system has been experimentally clarified.

The obtained results can be summarized as follows:
1. Ar/He mixture gas improves spheroidization process through the enhanced plasma enthalpy and heat transfer compared with pure argon gas.
2. Low particle velocity strongly affects the improvement of spheroidization rate.
3. Spheroidization rate drastically increases from 48.8 % to 82.7 % with as small as 4 % of helium content in central gas through the enlargement nozzle of diameter.

References