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Effect of gas flow rates and nozzle throat width on deposition of α-alumina films of granule spray in vacuum

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ABSTRACT

Deposition of α-alumina film by spraying alumina granules in a vacuum at room temperature was performed using a modified aerosol deposition system that had a supplemental gas flow line in addition to the carrier gas flow line. The pressure difference between the nozzle inlet and deposition chamber was increased by increasing the supplemental gas flow rate or by decreasing the nozzle throat width. For the same nozzle, the alumina film thickness per granule consumption increased as the pressure difference was increased. However, the film obtained by using the wide throat nozzle was thicker than that by using the narrow throat nozzle in spite of the smaller pressure difference except for the case without using the supplemental gas. The deposition behaviors according to the supplemental gas flow rate and the nozzle throat width were explained in part by the friction-affected layer near the nozzle throat wall and a critical granule velocity for film deposition. Alumina granules and films were characterized by scanning electron microscopy, X-ray diffractometry, surface profilometry, and transmission electron microscopy to assess and correlate the film quality to the deposition conditions.

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1. Introduction

Granule spray in vacuum (GSV) is a method for deposition of dense nano-structured ceramic film at room temperature by spraying granules that are flowable agglomerates of fine ceramic particles [1]. It was developed from the aerosol deposition method (AD) [2–4] for providing long-term stability of material feeding to a nozzle. Unlike other deposition methods of ceramic films using ceramic particles including thermal spray [5] and screen printing [6], both GSV and AD do not require an external heat source during or after deposition. The ceramic films deposited by GSV or by AD exhibited higher density (>95% theoretical density [2]) than those deposited by conventional thermal spray or by screen printing techniques. The only energy supplied for deposition in GSV and AD is the kinetic energy of the particle. It is this kinetic energy that is thought to facilitate the fracture and plastic deformation of the particles upon impact with the substrate. Therefore, it is commonly believed in these processes that the deposition of a particle requires a minimum critical velocity to undergo the necessary deformation, fracture, and bonding to the substrate or the pre-existing film [1–4].

As well documented in the literature [1–4,7], there are two chambers in deposition system of GSV or AD. The first chamber contains granules or powder, and the second chamber (called as deposition chamber) is evacuated by vacuum pumps. The first chamber is connected to the deposition chamber by means of a tube that feeds into a spray nozzle inside the deposition chamber. During the deposition process in AD, a gas flows into the first chamber (called as aerosol chamber) and carries particles into deposition chamber via the spray nozzle. Upon entering the spray nozzle, the gas and entrained particles gain substantial kinetic energy by pressure difference between the two chambers [2–4]. The particles ejected from the nozzle impact the substrate inside the deposition chamber. The particle impact velocity was increased as the gas flow rate was increased as reported in Ref. [2,8,9].

The spray nozzle used in AD or GSV is considered as one of the important variables affecting the pressure difference between the above two chambers and the deposition behavior of the film. Akedo and Lebedev reported that the particle flow velocity was higher for the converging type nozzle with small orifice than for the nozzle with big orifice [9]. Lee et al. found that their specially designed

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Table 1
Sample names and the amounts of granules used.

Nozzle throat width (mm)		Air flow rate ($\times 10^{-5}$ m^3/s)					
		Carrier 8.3	Supplemental 0	Carrier 8.3	Supplemental 17	Carrier 8.3	Supplemental 33
Sample name	0.8	0.8–8.3/0		0.8–8.3/17		0.8–8.3/33	
	0.4	0.4–8.3/0		0.4–8.3/17		0.4–8.3/33	
Amount of granules		150 g		90 g		90 g	

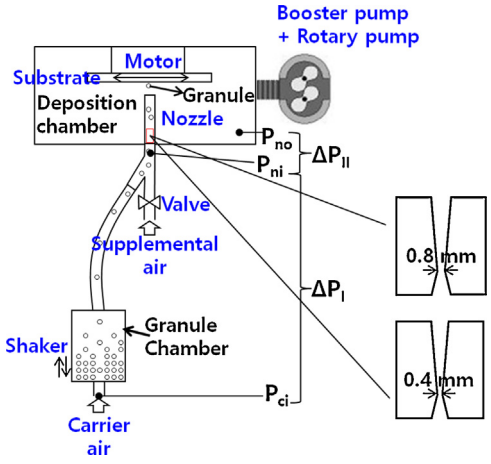

Fig. 1. Schematic diagram of the deposition system.

converging type nozzle with small orifice was effective for growing the alumina film fast on a hard substrate like sapphire by AD mainly due to the increased particle velocity [10]. Therefore, it is understood that the particle velocity was increased by decreasing the orifice size of the nozzle probably due to the increased pressure difference between the aerosol chamber and deposition chamber. In this work, we explored the deposition parameters and characteristics of alumina films using three different gas flow rates with two different nozzles in terms of the throat width.

2. Materials and methods

2.1. Pressure measurements

Fig. 1 shows a schematic diagram of the deposition system used for this study. The two converging-diverging type nozzles used for this study are also shown in the drawing. The two nozzles used had identical dimensions except for the throat width; 35×0.8 mm^2 and 35×0.4 mm^2, referred to in the text as 0.8 and 0.4 nozzles, respectively. Unlike the typical manner of supplying gas in AD where only the single carrier gas line was used [2,4,7–11], in this study, two separate gas lines, i.e., the carrier gas and the supplemental gas, were used. Medical grade purified air was used as the carrier gas and the supplemental gas. Two mass flow controls (Model 3665, Kofloc, Kojima Instruments Inc., Kyoto, Japan) were employed for controlling the carrier air flow rate and the supplemental air flow rate independently.

The carrier air directed into the granule chamber facilitated carrying the granules to nozzle. The supplemental air that joined near the nozzle acted as a further accelerator by increasing the total flow rate and pressure on the inlet side of the nozzle. Pressure was measured at the three points indicated in Fig. 1 during air flowing without granules by using a low vacuum gauge (KVC400-S5, KVC, Bucheon, Korea) at the inlet of the granule chamber (P_{ci}), at the inlet of the nozzle (P_{ni}) and within deposition chamber (P_{no}). The flow rate of the carrier air was fixed at 8.3×10^{-5} m^3/s for this study. The supplemental air flow rate was set at 0, 1.7, and 3.3×10^{-4} m^3/s. For the case where the supplemental air was not used (0 flow rate), a valve placed between the mass flow control and the nozzle inlet was closed.

Table 2
Deposition variables for making alumina film by GSV.

Variables	Value
Carrier air flow rate	8.3×10^{-5} m^3/s
Supplemental air flow rate	0, 1.7×10^{-4} m^3/s, 3.3×10^{-4} m^3/s
Nozzle throat dimensions	35×0.8 mm^2, 35×0.4 mm^2
Stand-off distance	10 mm
Substrate traveling distance	37 mm
Substrate traveling speed	1.67 mm/s
Film area	20×12 mm^2

2.2. Granule preparation

Commercially available α-alumina powder (AL-160SG3, Showa Denko, Tokyo, Japan) was used. Average particle size of the powder (d_{50}) measured by a particle size analyzer (LS 13320, Beckman Coulter, Inc., Fullerton, CA, USA) was 0.84 μm. The crystalline phase of the powder was analyzed by an X-ray diffractometer (D/Max 2200, Rigaku, Tokyo, Japan), and the obtained XRD pattern matched well to the α-alumina (JCPDS card number 10-173).

The α-alumina powder was granulated by spray drying at Dongjin Technology Institute, Ansan, Korea. Granules were heated to 973 K for four hours in air to remove the organics added for spray drying. There was a 3.5 wt% decrease after the heat treatment that corresponded well to the organic additive content information from Dongjin Technology Institute. After the heat treatment, granules were passed through a sieve with a 355 μm mesh. Granules were observed by using SEM (JSM 5800, Jeol, Tokyo, Japan) and images of five different areas under the same magnification were used for granule size measurements. A total of 1207 granules were measured by using image analysis software (ImagePro Plus 6, Media Cybernatics Inc., Rockville, MD, USA).

2.3. Alumina film deposition

For deposition of alumina films, granules after the heat treatment were poured in the granule chamber shown in Fig. 1. The amount of granules used for preparing each sample is shown in Table 1. Table 1 also shows the sample names according to the deposition conditions. A slide glass with dimensions of $25 \times 75 \times 1$ (thickness) mm^3 was used as a substrate and taped to a motor stage in the deposition chamber, as shown in Fig. 1. Part of the substrate was masked with scotch tape to expose 20×12 mm^2 film area in order to facilitate measurement of the film thickness after deposition. Details of the deposition variables are shown in Table 2.

After the deposition system was evacuated by a rotary pump and a booster pump to 6.5 Pa, the motor stage was powered on and both carrier air and supplemental air were started at the same time. Immediately after the air started to flow, a shaker connected to the granule chamber was activated at a slow speed just enough

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