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MICROSTRUCTURE AND PORE CHARACTERISTICS OF A SUS316L GAS FILTER FABRICATED BY WET POWDER SPRAYING

In this study, a flake-shaped metal powder was coated on a tube shaped pre-sintered 316L stainless steel support using a wet powder spraying process to fabricate a double pore structure, and the pore characteristics were analyzed according to coating time and tube rotation speed. The thickness of the coated layer was checked via optical microscopy, and porosity was measured using image analysis software. Air permeability was measured using a capillary flow porometer. As a result of the experiment, the optimal rotation speed of the support tube was established as 200 rpm. When the rotation speed was fixed, the coating thickness and the coating amount of the double pore structure increased as the coating time increased. The porosity of the double pore structure was increased due to the irregular arrangement of the flake-shaped powder. The air permeability of the double pore structure decreased with increasing fine pore layer thickness.

Keywords: Wet powder spraying process; Double pore structures; Pore properties; Rotation speed; Coating time

1. Introduction

With the steady development of personal computer and smartphone technology since the 1980s, the amount of research on core components of the semiconductor manufacturing process has grown exponentially. Also, as the degree of integration of semiconductor devices has increased, circuit line widths have become finer, and accordingly, an ever higher purity of materials used in the manufacturing process is required. In particular, the purity of the gas used in the semiconductor manufacturing process determines the productivity and performance of the semiconductor device [1]. In order to supply a high-purity process gas in the semiconductor manufacturing process, particles that may be included in the gas must be removed and the high-purity gas must be supplied. Therefore, it is important to develop an effective gas filter for use in semiconductor manufacturing [2,3].

Gas filters made of polymers and ceramic materials have limited usage environments due to the inherent properties of these materials. Metal filters, on the other hand, can be applied in high-temperature, high-pressure, and corrosive environments [3]. Metal filter manufacturing methods include uniaxial pressing, cold isostatic pressing, and metal injection molding using metal fiber [4] and metal powder [5,6]. When a metal filter is manufactured using metal fibers, there is a disadvantage in that the pore structure is very simple and expensive. In contrast, a metal filter manufactured using metal powder has excellent filtration ability due to its complex pore structure. However, pressure loss is relatively high. Therefore, it is necessary to manufacture a metal powder filter with high filtration efficiency and low pressure drop. There have been attempts to manufacture a gas filter having a double pore structure that includes coarse pores and fine pores by using a metal powder that can be used at high temperatures and which has excellent corrosion resistance. The material SUS316L has excellent corrosion resistance and is a widely used material for semiconductor process gas delivery components.

In this study, the objective was to fabricate a metal filter with a high filtration efficiency and a low pressure drop by double pore structure of fine pores and coarse pores using a wet powder spraying (WPS) process [7]. Flake-shaped SUS316L powder was coated on the outer surface of a tube-shaped support made of irregularly-shaped SUS316L powder using WPS, and the pore characteristics according to tube rotation speed and coating time were studied.

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2. Experimental

This research used a tube shape filter (Φ30 mm × 70 mm) with coarse pores fabricated using cold isostatic pressing (CIP) of the SUS316L powder manufactured by water atomizing as support. The CIP process involved loading the powder into the mold, charging the mold filled with the metal powder into the equipment, pressurizing the mold at a high pressure of 1,000-3,000 bar, and then removing the molded body from the mold. Using CIP, a support having a porosity of about 25-35% could be prepared.

Fig. 1 shows the flake-shaped powder and the tube-shaped support as well as, a schematic diagram of the WPS process using metal powder slurry. The WPS process is a technology that sprays slurry onto a tube support using a spray gun, allowing multiple tubes to be coated in just one spray. In this research, as reflected in TABLE 1, an experiment was conducted that included slurry flow rate and air pressure as fixed variables.

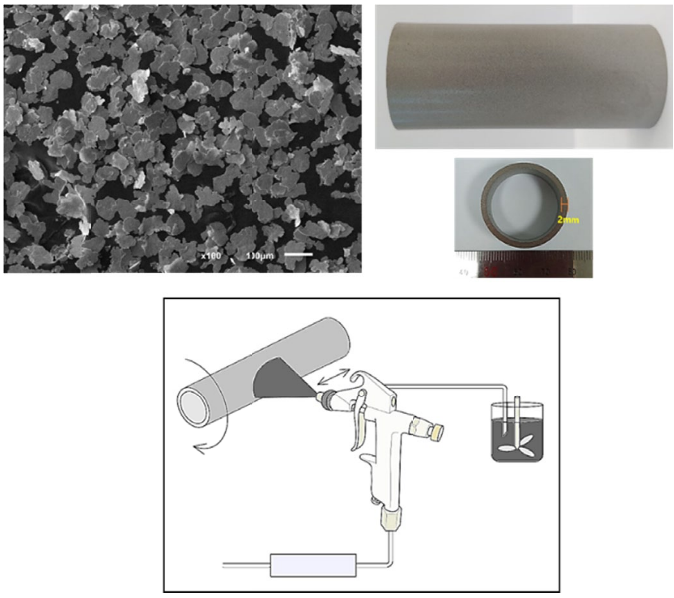


Fig. 1. Schematic diagram for the fabrication of metal powder filter using wet powder spraying

TABLE 1

Wet powder spraying experiments conditions

Support	Exterior diameter (30 mm), Inner diameter (25.5 mm), Length (70 mm)
Spray nozzle type	Vortex (Atomax BN160)
Binder	MC 8 wt.% + Distilled water 92 wt.%
Slurry (wt.%)	Powder 40 + Binder solution 20 + Ethanol 40
Slurry flow rate (ml/min)	200
Air pressure (MPa)	0.09
Distance between nozzle tip and tube (mm)	270
Width of movement (mm)	350
Nozzle moving speed (mm/sec)	299.15

The tube rotation speed was varied (150, 200, and 250 rpm), and the coating thickness was checked to find the optimal rotation speed. After fixing the rotation speed, the coating time was changed to 35.1, 58.5, and 81.9 sec, and the coating amount, coating thickness, porosity, and permeability were measured. The coating thickness of the metal powder filter was measured with an optical microscope (Nikon ECLIPSE MA200, JAPAN), and the porosity was measured using an image analysis program (iSolution DT x64) that took optical microscope images for its source data. In addition, the measurement of air permeability was analyzed with a capillary flow porometer (CFP1200AEL, PMI, USA).

3. Results and discussion

First, as shown in the cross-sectional structures of Figs. 2 and 3, it can be confirmed that a metal powder filter having a double pore structure was made by using the WPS process. Also, it can be seen that the pore size of the fine porous layer is smaller than that of the coarse porous layer and forms a more complex flow path. In order to form a uniform fine porous layer on the surface of the tube-shaped support, the support should be rotated. If the tube rotation speed is too fast, the metal powder slurry sprayed through the spray nozzle may bounce off and fail to coated. Therefore, it is important to find an appropriate rotation speed according to the metal powder slurry being sprayed.

Fig. 2 shows the microstructure of a metal powder filter having a double pore structure according to tube rotation speed. It can be seen that when the rotation speed of the tube increases from 150 rpm to 200 rpm, the thickness of the fine porous layer increases from 154.76 μm to 179.89 μm and then decreases to 124.17 μm at a rotation speed of 250 rpm. As expected, if the tube rotation speed is as high as 250 rpm, the coating thickness formed will be thin because the amount of powder that bounces off increases when the slurry sprayed through the spray nozzle is coated on the surface of the support. Subsequently, looking at the correlation between the coating amount and the coating thickness according to the tube rotation speed, as seen in the microstructure, 2.06 g is coated on the support when coating is performed at a speed of 150 rpm, and 2.14 g is coated on the support when coating is performed at a speed of 200 rpm. And when coating at a speed of 250 rpm, 1.63 g is coated on the support. It was confirmed that coating amounts increased between 150 rpm and 200 rpm, but when coating at a speed of 250 rpm, the amount of coating becomes smaller and the coating thickness becomes thinner because more powder has bounced off. Therefore, it was found that the coating should be performed at an appropriate tube rotation speed during the WPS process. Based on these results, subsequent experiments were conducted for other variables while rotating the support at the optimal tube rotation speed (200 rpm).

Fig. 3 shows the microstructure, coating amount, and coating thickness change of the metal powder filter having a double pore structure according to coating time. As the coating time

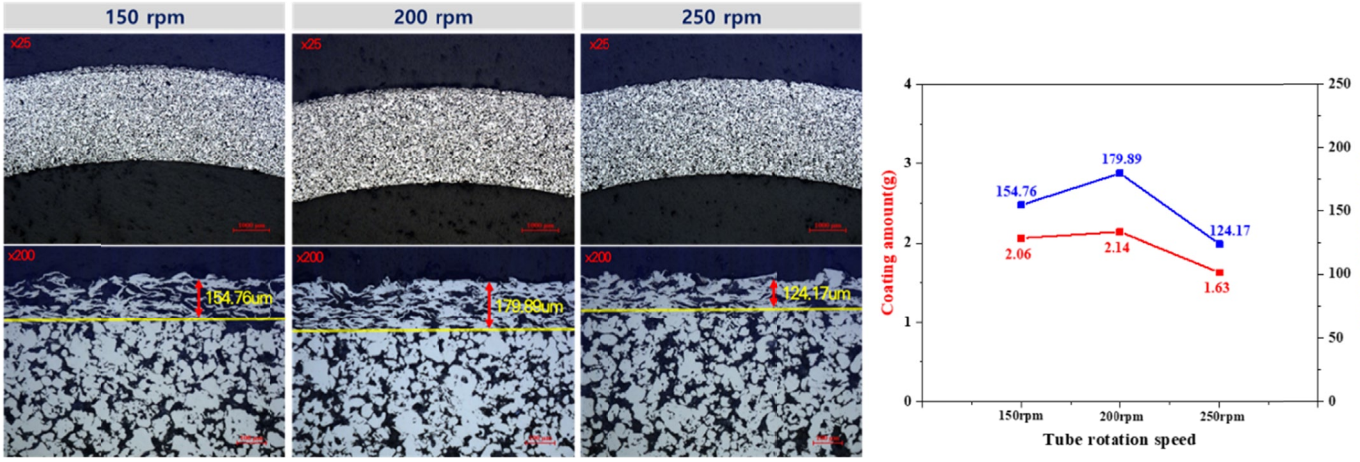


Fig. 2. The cross-sectional structure, coating amount and coating thickness of the metal powder filter according to tube rotation speed

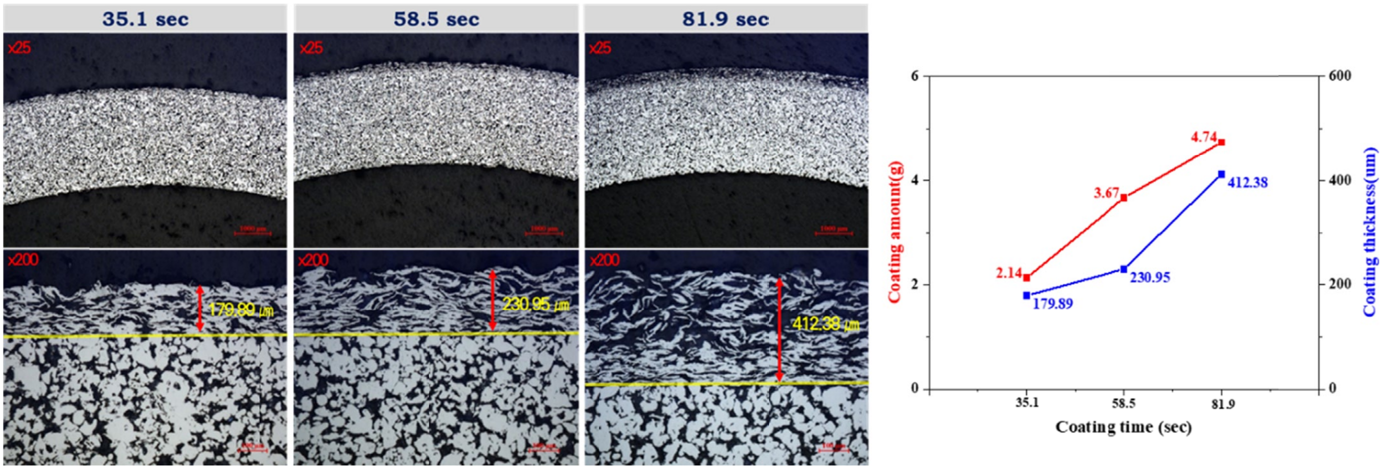
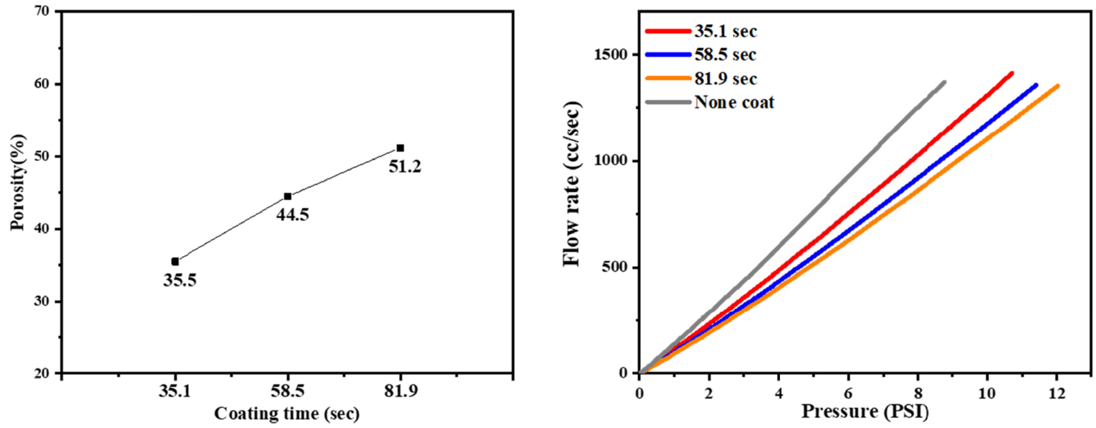


Fig. 3. The cross-sectional structure, coating amount and coating thickness of the metal powder filter according to coating time

increases to 35.1 sec, 58.5 sec, and 81.9 sec, the coating amount increases to 2.14, 3.67, and 4.74 g, respectively, and it can be seen that the coating thickness also increases to 179.89, 230.95, and 412.38 μm.

Fig. 4 shows the porosity and air permeability characteristics of the support manufactured using the CIP and the metal powder filter having a double pore structure manufactured by coating the flake powder on the support using the WPS process. It shows that the porosity increases from 35.5% to 51.2% as the coating time increases from 35.1 sec to 81.9 sec. This seems to be because when the flake-shaped powders are spray-coated in the vertical direction to the support surface during the WPS process,



(a) (b)

Fig. 4. The pore characteristics of the metal powder filter according to coating time

the flake-shaped powders are arranged in an irregular direction rather than parallel to the surface of the support. In addition, it can be seen that the support having a single coarse porous layer has the highest air permeability, and as the WPS coating time increases, the thickness of the fine porous layer increases and the air permeability decreases, but unlike the increase in porosity it does not decrease dramatically.

4. Conclusions

In this study, WPS was performed with three different tube rotation speeds, and the optimal rotation speed was found by checking the coating thickness. If the rotation speed was too fast, the metal powder slurry sprayed through the spray nozzle could bounce off and fail to leave an adequate coating, so the appropriate rotation speed was selected as 200 rpm. After fixing the rotation speed, the coating thickness and the coating amount of the double pore structure increased as the coating time increased. The porosity of the double pore structure was increased by the irregular arrangement of the flake-shaped powder. The air permeability of the double pore structure decreased with increasing fine porous layer thickness. In this study, it was found that by coating the flake-shaped powder using WPS process, it was possible to fabricate a metal powder filter with high porosity without significantly reducing air permeability, that is, with a low pressure drop and high filtration efficiency.

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