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FABRICATION OF A PLATE-TYPE POROUS STAINLESS STEEL 316L POWDER FILTER WITH DIFFERENT PORE STRUCTURES WITHOUT USE OF A BINDER

Generally, when fabricating porous filters using metal powder, about 1 to 2 wt% of a binder is added to increase the formability of the metal powder. If the binder is not completely removed through a debinding and sintering process, however it can cause defects such as discoloration and the generation of fine particles. In this study, a study was conducted to fabricate a plate-type porous stainless steel powder filter with a different pore structure without the use of a biner. First, the metal powder is charged into a ceramic mold to form a plate-shaped powder filter, and then covered with a ceramic top plate. In order to observe the pore properties according to the pressure, the unpressurized specimen and the specimen pressurized with a pressure of 30 MPa were then sintered at 1050°C for one hour in a high vacuum atmosphere furnace. The microstructure of the sintered plate-shaped powder filter was observed through an optical microscope and in order to analyze its pore properties as a filter, gas permeability and porosity were measured using a capillary flow porometer and Archimedes' law.

Keywords: Gas Filter; membrane; Stainless steel; Porosity; Powder shape

1. Introduction

Many types of gases are used in semiconductor manufacturing processes such as epitaxy, etching, cleaning, ion implantation, doping, annealing, passivation, blanketing, and so on. These gases are transported from gas storage facilities through gas purifiers and gas distributors to the semiconductor device production process [1]. The metal filter for semiconductor equipment is a key component that serves to protect the inflow of impurity particles that may exist in the process gas used in the semiconductor device production process [2]. Ultrafine particles are a major cause of pattern defects in semiconductor processes, and metal filter components to reduce them in highly integrated semiconductor processes are becoming increasingly important as an indispensable element. Adequate filtration performance of nano-ultrafine particles and lower pressure loss characteristics of the front and rear side of the filter are thus required [3]. The metal filter must have a pore structure that impedes fluid flow to improve the filtration performance and also increases the porosity so that the pressure drop is small. That is, it should have a low air permeability and relatively high porosity at the same time [4,5]. Methods of manufacturing a porous metal filter include semi-solid foaming, gas expansion, metal hollow sphere (MHS) sintering, a space holder technique, and slurry coating, among other methods [6-10]. As the pore sphere, pore size, porosity, and air permeability are characteristic factors of the porous metal that vary in accordance with the manufacturing method, the manufacturing method is selected according to the purpose of use. In particular, the binder, which is often mixed together for adhesion between particles in the forming process of the powder metallurgy, may act as a defect that causes discoloration of the final product or creates impurities if not completely removed during the heat treatment process [11]. In this study, in order to resolve these binder problems and produce a large size plate-type metal powder filter having low air permeability and relatively high porosity, a metal filter was manufactured using stainless steel powder having a flake-shape followed by sintering in a ceramic mold without the use of a binder.

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2. Experimental

In this study, three types of stainless steel 316L powders with different shapes and sizes are prepared. In the experiment, an irregular-shaped powder with a mean powder size of about 50 μm manufactured by a water atomization process (Daido, Japan) and a flake-shaped powder with mean powder sizes of 36 μm and 60 μm (Novamet, USA), respectively, were used. Fig. 1, shows the shape and powder size distribution of the metal powder used in this study. Before fabricating a plate-type porous metal filter, a mold with a size of 100×100 mm was prepared using alumina. Guide pins are fixed on the lower plate with a thickness of 10 mm, spacers with a thickness of 1.2 mm and a hole size of 100×100 mm are stacked, and then metal powder is evenly charged. About 40 g of the irregular powder, and 15~16 g of the flake-shaped powders are respectively employed. When the spacer thickness was 1.2 mm, the amount of powder used in during the process was controlled differently depending on the particle size and shape of the powder. The powder is then charged into an alumina mold and then flattened. During subsequent high-vacuum sintering, a punch-shaped upper plate is covered to prevent powder scattering. For comparison of the pressurization of the powder compacts, non-pressurization and pressurization processes are performed. In the case of pressurization, pressure of 30 MPa is applied with a laboratory hydraulic press for 30 seconds. The powder compacts with the ceramic molds are sintered at 1,050°C for one hour in a high vacuum ($<5\times10^{-5}$ torr). The fabricated plate-type porous stainless steel powder filter is evaluated for its characteristics as a filter by measuring the microstructure through an optical microscope (ECLIPSE MA200, Nikon, Japan) and air permeability using a capillary flow porometer (CFP1200AEL, PMI, USA).

3. Results and Discussion

Fig. 2 shows photographs of plate-type porous stainless steel filters with a size of about 100×100 mm having a single pore structure fabricated using the irregular-shaped powder and the flake-shaped powder. When fabricated with the flake-shaped powder, it can be seen that the surface is remarkably smoother than that fabricated with the irregular-shaped powder. Fig. 3 shows the microstructure of the plate-type porous stainless steel powder filter according to the type of powder with and without pressurizing. All the pressurized specimens show a denser microstructure than the non-pressurized specimens, and the powder is arranged in a direction perpendicular to the pressing direction in the specimen fabricated with the flake-type powder. In addition, the plate-type porous stainless steel filter made with the irregular-shaped powder decreases in thickness to about 1.2 mm in the case of non-pressurization and 1.08 mm in the case of pressurization. The thickness decreases slightly when pressing is performed from 760 μm to 550 μm for the flake-shaped powder with an average powder size of 36 μm and from 720 μm to 470 μm for the flake-shaped powder with an average powder size of 60 μm. The thickness decreases due to the reduction of voids between the particles as a result of the application of pressure. Figs. 4 and 5 shows the porosity and air permeability characteristics of the plate-type porous stainless steel powder filter depending on the type of powder with and without pressurizing. The porosity was 66.7% for the irregular-shaped powder, 77.8% for the flake-shaped powder with a particle size of 36 μm, and 75.9% for the flake-shaped powder with a particle size of 60 μm when a non-pressurized specimen was used. As can be seen from the microstructure above, the overall porosity tends to decrease as the pressure is applied. In addition, the porosity is higher in the

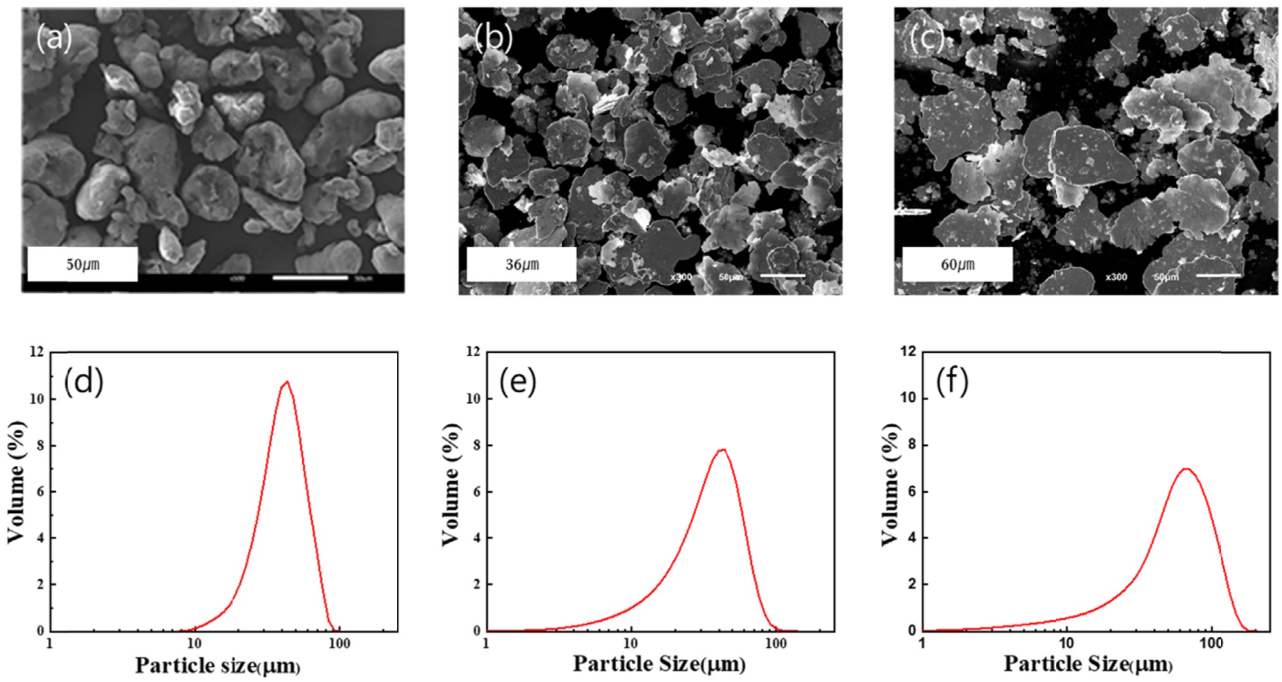
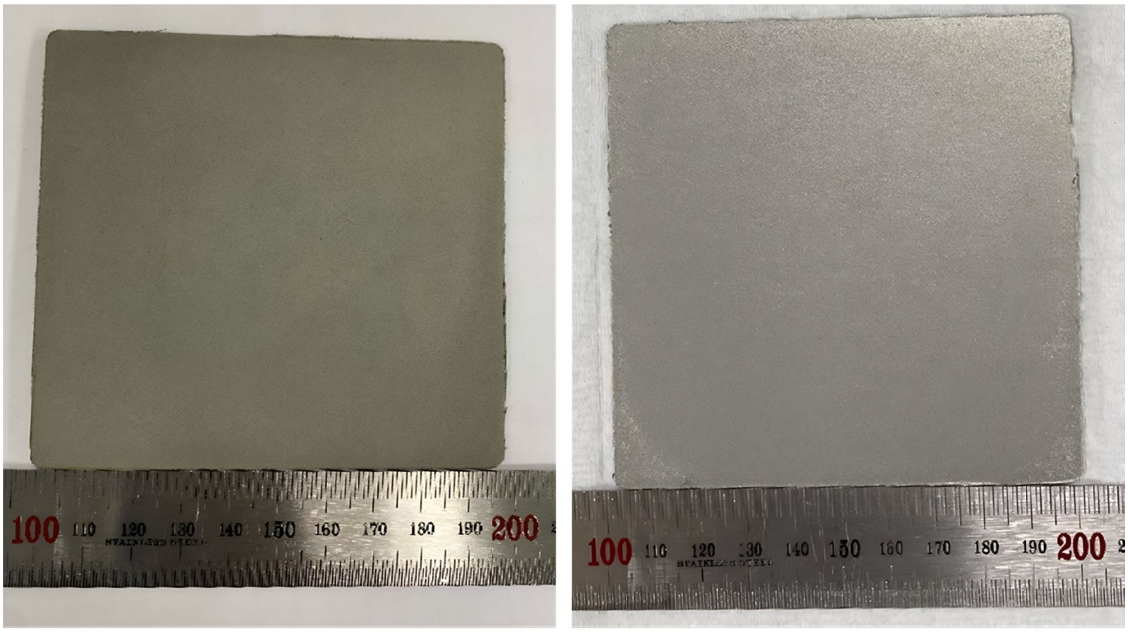


Fig. 1. SEM image of the stainless steel 316L powder morphology and powder size distributions (a), (d) irregular-shape powder, (b), (e) flake-shaped powder (36 μm), (c), (f) flake-shaped powder (60 μm)



(a) Irregular shape powder (b) Flake shape powder

Fig. 2. Photograph of the plate-type powder filter fabricated with (a) irregular-shaped powder and (b) flake-shaped powder

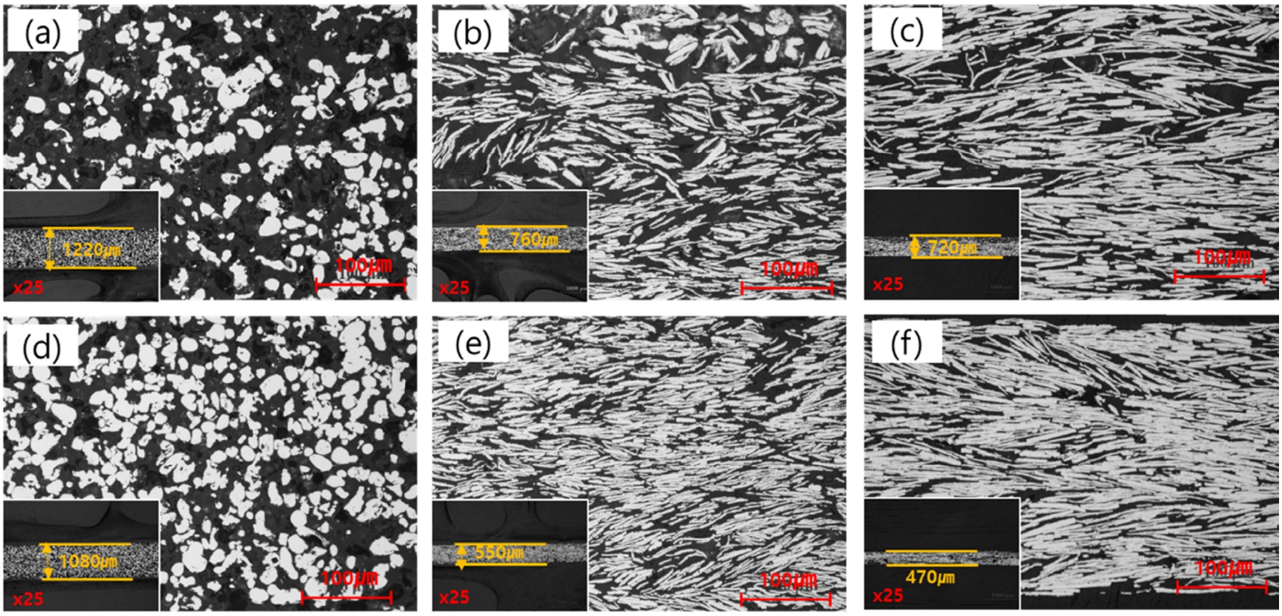


Fig. 3. Cross-sectional image of non-pressurized specimens fabricated with (a) irregular-shaped powder (b) flake-shaped powder (36 μm) and (c) flake-shaped powder (60 μm) and cross-sectional images of the pressurized specimens fabricated with (d) irregular-shaped powder (e) flake-shaped powder (36 μm) and (f) flake-shaped powder (60 μm)

case of using the flake-shaped powder than in the specimen fabricated with the irregular-shaped powder. This is attributed to the tap density of the flake-shaped powder being lower than that of the irregular-shaped powder. As can be expected from the microstructure and porosity results, the air permeability is lower in the case of pressurization in general, and the air permeability of the specimens fabricated with the flake-shaped powder is generally lower than that of the specimens made with the irregular-shaped powder. In addition, in the case of the specimen fabricated using the large powder having an average powder size of 60 μm, the porosity and permeability are lower than those of the specimen fabricated with the powder having an average powder size of 36 μm. The cause of this phenomenon is ascribed to the shape of the powder. In the specimens with the flake-shaped powder with a large diameter and thin thickness, the flake-shaped powders are arranged in a direction perpendicular to the flow of air. This pore structure can interfere with the flow of air, and eventually the air permeability is lowered compared to the irregular-shaped powder. As mentioned before, in order to use filters for semiconductor applications, they should have a high porosity and low air permeability. In this study, it is demonstrated that filters using a flake-shaped powder with a porosity of over 75% that still exhibited low air permeability characteristics could be manufactured.

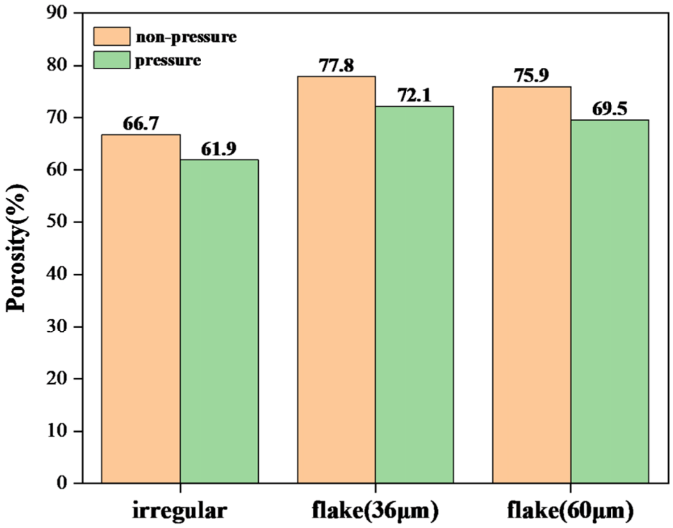


Fig. 4. Porosity of the plate-type powder filter fabricated with different powder and pressurizing

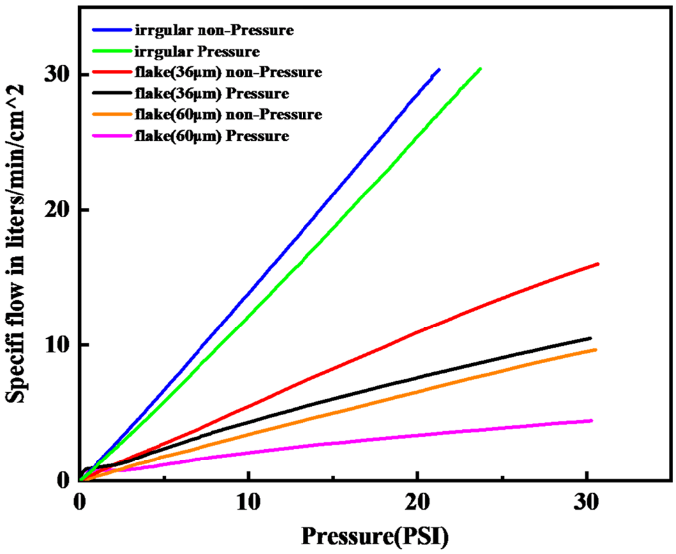


Fig. 5. Air permeability of the plate-type powder filter fabricated with different powder and pressurizing

4. Conclusion

In this study, a 100×100 mm size of plate-type porous stainless steel powder filter was fabricated without using a binder. Flake-shaped powder and irregular-shaped powder with different sizes were prepared by applying variables according to the size and shape of the powders and pressurization, respectively. The porosity tended to be higher in the case of using the flake-shaped powder than in the specimen fabricated with irregular-shaped powder. In the specimens fabricated using flake-shaped powders, it can be seen that the porosity increases as the average powder size decreases and the air permeability decreases as the average powder size increases. In conclusion, it is possible to fabricate a plate-type porous stainless steel powder filter using flake-shaped powder that has a high porosity and low air permeability. In the future, a plate-type porous stainless steel powder filter having a double-pore structure will be fabricated.

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REFERENCES

[1] Heikkinen, Maire SA, Naomi H. Harley, Experimental investigation of sintered porous metal filters. J. Aerosol. Sci. **31**, 6, 721-738 (2000). DOI: https://doi.org/10.1016/S0021-8502(99)00550-9

[2] Bo Zhu, et al. Short review on porous metal membranes – Fabrication, commercial products, and applications. J. Membr. Sci. **8** (3), 83 (2018). DOI: https://doi.org/10.3390/membranes8030083

[3] Ryi. Shin-Kun, et al., Development of a new porous metal support of metallic dense membrane for hydrogen separation. J. Membrane Sci. 439-445 (2006). DOI: https://doi.org/10.3390/membranes8030083

[4] Li. Ming, et al., Metal binder jetting additive manufacturing: a literature review. J Manuf Sci E-T ASME. **142** (9), 090801 (2020). DOI: https://doi.org/10.1115/1.4047430

[5] S. Uemiya, State of the art of supported metal membrane for gas separation. Sep. Purif. Methods. **28** (1), (1999). DOI: https://doi.org/10.1080/03602549909351644

[6] Nam-Hoon Kim, et al., Fabrication and Characterization of Porous Nickel Membrane for High Precision Gas Filter by In-situ Reduction/Sintering Process. J. Korean Cryst. Growth Cryst. Technol. **16** (4), 262-267 (2009). DOI: https://doi.org/10.4150/KPMI.2009.16.4.262

[7] S.K. Ryi, et al., Fabrication of nickel filter made by uniaxial pressing process for gas purification: Fabrication pressure effect. J. Membrane Sci. **299**, 1-2, 174-180 (2007). DOI: https://doi.org/10.1016/j.memsci.2007.04.042

[8] D. Park, et al., Fabrication and Pore Characteristics of Cu Foam by Slurry Coating Process. J. Korean Powder Metall. Inst. **22**, 2, 87-92 (2015). DOI: https://doi.org/10.4150/KPMI.2015.22.2.87

[9] D.W. Schaefer, Engineered Porous Materials. Mrs Bull. **19**, 14-19 (1994). DOI: https://doi.org/10.1557/S0883769400039452

[10] L. Zhao, et al., Casting solvent effects on morphologies, gas transport properties of a novel 6FDA/PMDA–TMMDA copolyimide membrane and its derived carbon membranes. J. Membrane Sci. **244**, 1-2 (2004). DOI: https://doi.org/10.1016/j.memsci.2004.07.005

[11] Gupta, Ajay, et al., Effect of humidity and particle hygroscopicity on the mass loading capacity of high efficiency particulate air (HEPA) filters. J. Aerosol. Sci. **19**, 94-107 (1993). DOI: https://doi.org/10.1080/02786829308959624