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EFFECT OF POWDER SHAPE AND SINTERING TEMPERATURE ON THE PREPARATION OF Ni-BASED POROUS METAL

Usually porous metals are known as relatively excellent characteristic such as large surface area, light, lower heat capacity, high toughness and permeability for exhaust gas filter, hydrogen reformer catalyst support. The Ni alloys have high corrosion resistance, heat resistance and chemical stability for high temperature applications. In this study, the Ni-based porous metals have been developed with Hastelloy powder by gas atomization and water atomization in order to find the effects of powder shape on porous metal. Each Hastelloy powder is pressed on disk shape of 2 mm thickness with 12 tons using uniaxial press machine. The specimens are sintered at various temperatures in high vacuum condition. The pore properties were evaluated using Porometer and microstructures were observed with SEM.

Keywords: Hastelloy, porosity, porous metal, powder shape, sintering temperature

1. Introduction

The term of porous metal refers to a structure in which many small cells are regularly or irregularly dispersed inside and are composed of 15 to 95% of the volume of pores. The porous metal has a large surface area and functional characteristic such as energy absorption capability and small thermal or electric conductivity [1]. Such a porous metal has been widely used in applications such as shock absorbers, filter, heat exchangers and so on [2]. Along with this, the porous metal can be used as a filter by using a large specific surface area and recently, it is expanding its application field. For a filter, the filtration performance of the porous material can be improved by increasing the area of absorbing and adsorbing contaminants depending on the finer pore size [3]. There are various methods of manufacturing the porous material such as a melt gas injection method, a molten metal foaming method, a reaction high metal foaming method, a metal vapor deposition method, and a space holder method, etc. [4,5]. These methods can be classified into a method using a molten metal and a method using a metal powder.

Particularly, when a porous metal is produced by using a powder metallurgy, it can be fabricated at a lower temperature than that of casting, and two or more kinds of powders can be uniformly mixed to easily produce an alloy of desired components and composition. When such a porous metal by powder metallurgy is used as a filter, it has been reported that the unburned solid particulate material can be collected internally and the back pressure to the filter can be small. However, since the manufacture of a porous metal by powder metallurgy is complicated, the production cost is high, and the mechanical properties of the finished product vary from the powder shape, particle size and particle size distribution of the powder, it should be chosen in accordance with purpose to do [6].

Methods for producing metal powders include mechanical methods, physical methods, electrochemical, chemical methods and atomization. Among them, the atomization is a process of dispersing molten metal into small droplets and then solidifying the particles into a particle form, which is capable of producing a powder having a fine particle size with high production speed, easy process control, also this method applies in polymers and ceramics. There are three main production methods of atomization: gas injection method, water (liquid) injection method, and centrifugal method. In the gas atomization method, spherical powder can be produced by impinging gas such as air, nitrogen, helium or argon on the droplets of the molten metal sprayed. In the water atomization method, instead of gas, water impact molten metal droplets and quench simultaneously, then the surface of produced powders shows a rough and irregular shape [7,9,10].

In this study, to research the effects of metal powder shape on the porous metal as a filter and catalyst carrier material, the gas-atomized powder and water-atomized powder were compressed to form disk type respectively. The metal powder used

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in this process is Hastelloy, a Ni-based alloy, which have strong corrosion and thermal resistance, as well as high chemical stability to be used at extreme conditions like high temperatures [8]. The pore properties and microstructure of the fabricated specimens were analyzed and through these results, we attempted to investigate the effects of powder shape.

2. Experimental

Fig. 1 shows a flow chart of a process for manufacturing of porous metal. In this study, Hastelloy, which is a Ni-based alloy, was prepared into a spherical powder by gas atomization and an irregular shape powder by water atomization. At first, 4.0 wt% PVA solution as a binder was mixed with Hastelloy powder to improve wettability and formability. Powder and binder were mixed at a weight ratio of 100: 3, and 5 g of Hastelloy powder was used per one specimen. A disk type specimen with a diameter of 25 mm was formed by applying a pressure of 12 ton for 30 seconds using a uniaxial pressing machine. In order to investigate the sintering behavior according to sintering temperature, three of pressing specimens were prepared by the spherical powder and the irregular shape powder respectively, and these were debinded in a tubular furnace to remove the organic binder of the compact specimens. As the debinding condition, the temperature was raised at a rate of 5°C/min in an Ar atmosphere (3 L/min) to prevent the oxidation of the alloy powder, and the furnace was kept at 550°C for 1 hour and then furnace-cooled. The debinded specimens were sintered at a maximum temperature of 1100°C, 1150°C, and 1200°C for 2 hours at a heating rate of 5°C/min in a high vacuum atmosphere (10^{-6} torr). The sintering temperature was set to a temperature under the melting point, which is about 1350°C to 1400°C, at which the surface of the powder particles can be melted. The porosity of the sintered specimens was measured by Archimedes method and the microstructure and phase analysis were performed using scanning electron microscope (SEM, JSM-6610LV, JEOL). The porosity and the mean pore size of the specimens were measured using a capillary flow porometer (CEP1200AEL, PMI, USA).

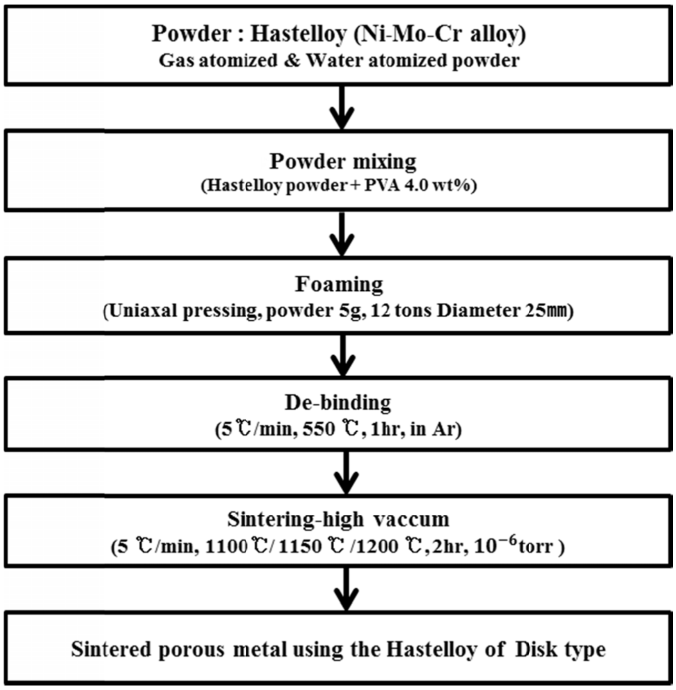

Fig. 1. Flow chart of fabrication process for sintered porous metal using the Hastelloy powders

3. Results and discussion

Fig. 2 is a SEM image of the Hastelloy powder produced by (a) gas atomization and (b) water-atomization. As mentioned in the previous section, the shape of the gas-atomized Hastelloy powder is spherical, and the shape of water-atomized Hastelloy powder is confirmed that the surface is very irregular.

Fig. 3 is a SEM image of sintered specimens at 1100°C, 1150°C and 1200°C after pressing the spherical powder and the irregular shape powder respectively. The higher the sintering temperature of each press specimen, the intergranular bonding shows more densification and the area of the pores is the smaller. As shown in (a), (b), and (c) in Fig. 3, it can be seen from the growth of the neck caused by the progress of bonding between particles. In the case of specimens sintered at (a) 1100°C, bonding

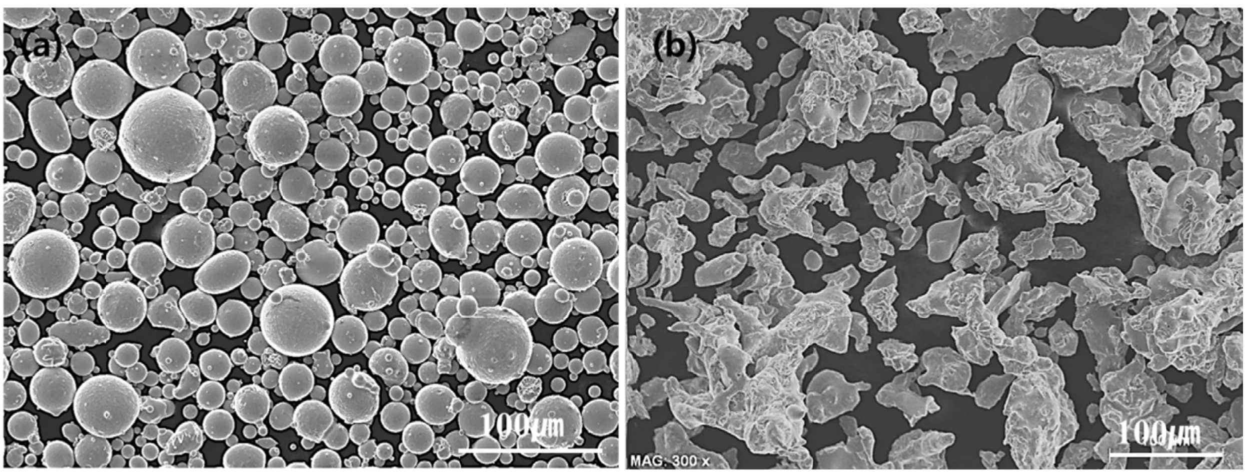

Fig. 2. SEM image of the Hastelloy powder morphology made by (a) the gas-atomizing process and (b) the water-atomizing process

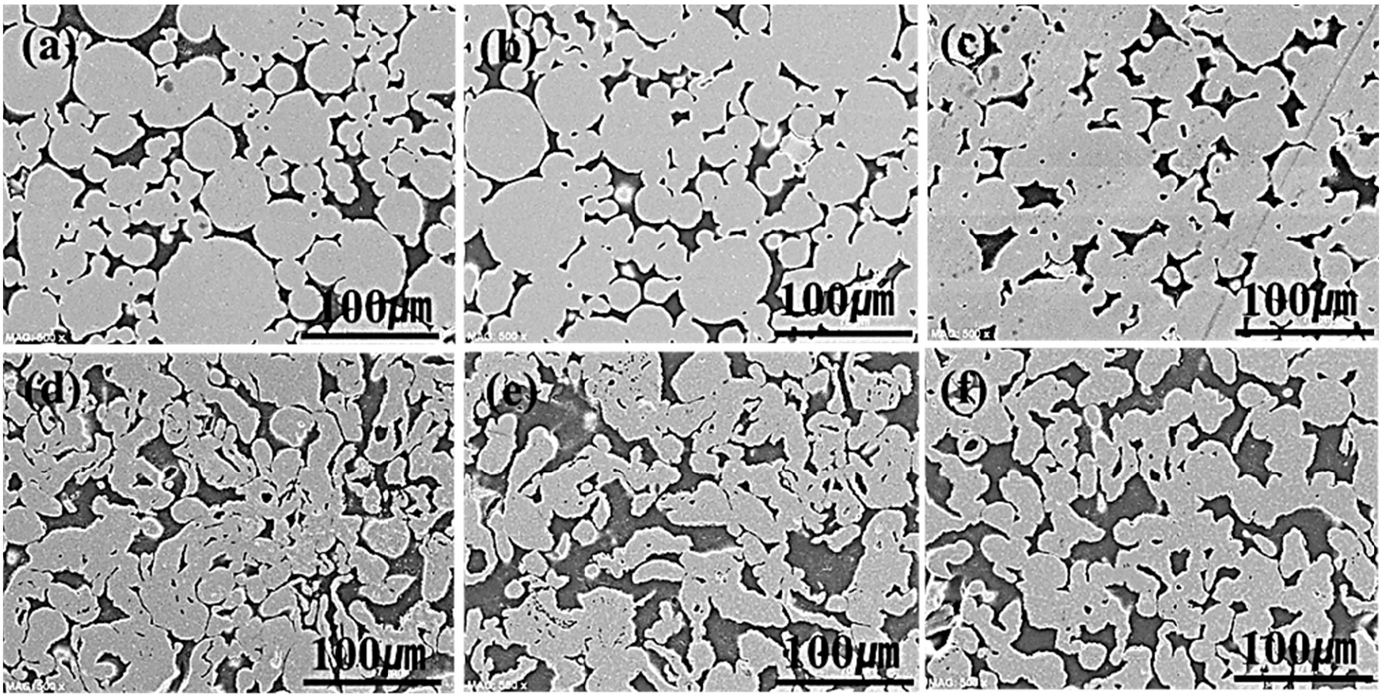

Fig. 3. SEM image of cross section about the gas-atomized Hastelloy powder sintered at (a) 1100°C, (b)1150°C, (c)1200°C, and the water-atomized Hastelloy powder sintered at (a) 1100°C, (b)1150°C, (c)1200°C

was progressed by contact between particles. At (b) 1150°C, the contact area between particles became broader. At (c) 1200°C, it is possible to observe many particles bonded together into one with the enlarged neck by broadened contact area.

In addition, in the case of pressed specimens of the irregular shape powder, it is possible to confirm a less compact structure than a pressed specimen of the spherical powder. As shown in (d), (e) and (f) in Fig. 3, it is considered that this is because the shape of the powder is so varied that a large pore of various shapes is formed, and then the size and shape of the pores are also very diverse.

Fig. 4 shows the gas permeability of the sintered specimens, it is compared of the specimens of the spherical powder with the irregular shape powder at different sintering temperature.

Both of the specimens can be confirmed to have lowered air permeability as the sintering temperature increases. This is in accord with the results of the analysis in Fig. 3, which shows that the higher the sintering temperature, the denser intergranular bonding. Compared both of the specimens shown in Fig. 4(a) and (b), the specimens of the irregular shape powder show relatively high air permeability as a whole. In Fig. 3, it shows that the pore between the irregular shaped particles have a larger area than the pore between the spherical particles. So, it is analyzed that the gas can flow more smoothly. In the case of Hastelloy specimens of the spherical powder, particles are more likely to block the connection between the pores, which can form isolated inner pores because the pore between the particles can be densely packed by a spherical shape having a relatively smooth surface.

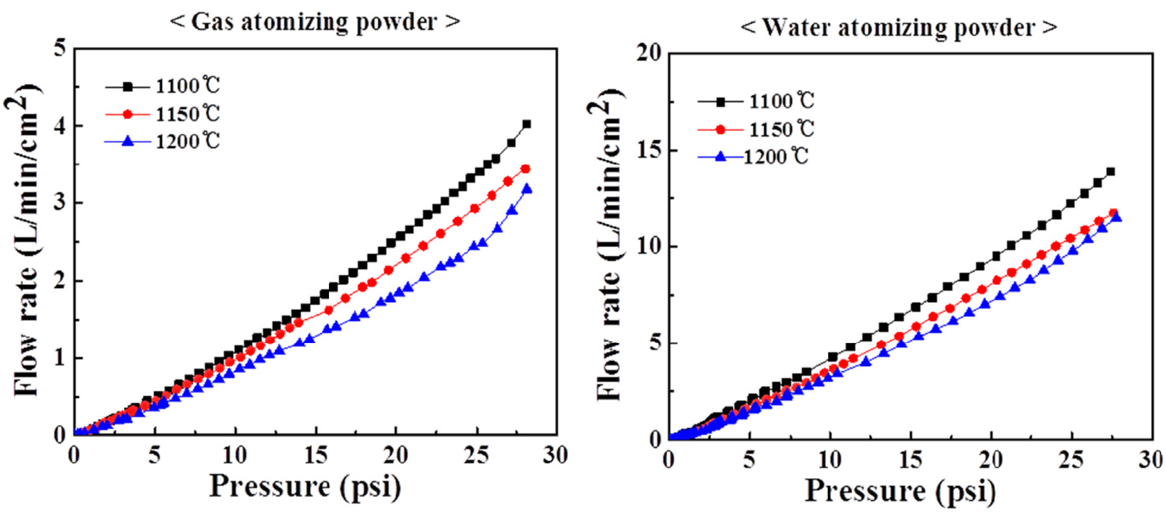

Fig. 4. Permeability of the porous metals made by (a) the gas-atomized Hastelloy powder and (b) the water-atomized Hastelloy powder

Therefore, it is expected that the formation of the penetrated pores and open pores, which are exposed to the outside of the body, is advantage for the irregular shape powder rather than the spherical shape powder.

Table 1 shows measured values of apparent porosity and mean pore size. The apparent porosity was calculated by comparing the volume density obtained by measuring the weight and volume of the specimen with the theoretical density of Hastelloy. Green is its measured porosity of the green body before sintering. The porosity of the green body by the spherical powder was 37.7% and as the sintering temperature increased from 1100°C to 1200°C, the porosities are 19.8%, 18.1% and 13.5%, respectively. The porosity of specimens by the irregular shape powder was 46.3% before sintering and 26.4%, 20.8% and 17.7% respectively according to increasing the sintering temperature. As shown in Fig. 4, the porosity decreases as the sintering temperature increases. Comparing the porosity of Hastelloy specimens of the spherical powder and that of the irregular shape powder, it can be confirmed that the specimen of the irregular shape powder have higher porosity.

TABLE 1

(a) Porosity and (b) mean pore size of Hastelloy porous metals made with the spherical powder and the irregular shape powder

	Porosity (%)		Pore size (μm)	
	Spherical	**Irregular**	**Spherical**	**Irregular**
Green	37.720	46.340	—	—
1100°C	19.832	26.441	0.69	2.79
1150°C	18.113	20.830	0.49	2.56
1200°C	13.503	17.711	0.39	1.57

The pore size shown was measured with a capillary flow porometer. The porous specimen was immersed in a Galwick solution (15.9 Dynes/cm) having a surface tension lower than that of water (72.0 Dynes/cm) and the pore size was measured by the amount of air passing through the specimen with gradually increasing the air pressure. The mean pore size of specimens of the spherical powder decreased from 0.69 μm, 0.49 μm to 0.39 μm with increasing sintering temperature of 1100°C, 1150°C and 1200°C. Also, the pore size of specimens of the irregular shape powder decreased from 2.79 μm, 2.56 μm to 1.57 μm as the sintering temperature increased. Comparing the pore sizes of both specimens manufactured by each powder, the specimen of the irregular shape powder has larger pore size, which is identical to the results analyzed in Fig. 3. In addition, specimen of the spherical shape powder has relatively small pore sizes, which contribute to the results of the measurements of relatively low air permeability and porosity. That is, when the shape of the powder particles is irregular, penetrated coarse pores are formed, and when the shape of the powder is spherical, many closed pores are formed inside.

However, the difference in permeability between the two specimens is large, but the difference in porosity is relatively small. This is because the air permeability is predominantly influenced by the pore size results. Also, the sintered density of the specimen influences the porosity. In this study, since both of specimen is fabricated by same amount of hastelloy powder, the difference in porosity is relatively small.

4. Conclusions

In this study, to investigate the effect of powder shape and sintering temperature on the fabricating of porous metal, we compared the spherical powder by gas atomization and irregular powder by water atomization with different sintering temperature. As the sintering temperature increased, the porosity of spherical powder by gas atomization decreased from 20% to 14% and the mean pore size was about 0.7 ~ 0.4 μm. The porosity of the irregular shaped powder by water atomization showed a relatively large reduction of porosity from 26% to 18% and the mean pore size of 2.8 ~ 1.6 μm. As a result, it was confirmed that the porosity and porosity of the porous metal can be changed according to powder shape, and the pore properties can be controlled according to the sintering temperature.

Acknowledgments

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