MIN-JEONG LEE*,**, YU-JEONG YI***, HYEON-JU KIM*, MANHO PARK***, BYOUNG-KEE KIM**, JUNG-YEUL YUN*#

A STUDY ON PORE PROPERTIES OF HASTELLOY POWDER POROUS METAL FABRICATED BY ELECTROSTATIC POWDER COATING

Porous metals show not only extremely low density, but also excellent physical, mechanical and acoustic properties. In this study, Hastelloy powders prepared by gas atomization are used to manufacture 3D geometries of Hastelloy porous metal with above 90% porosity using electrostatic powder coating process. In order to control pore size and porosity, foam is sintered at 1200~1300°C and different powder coating amount. The pore properties are evaluated using SEM and Archimedes method. As powder coating amount and sintering temperature increased, porosity is decreased from 96.4 to 94.4%. And foam density is increased from 0.323 to 0.497 g/cm³ and pore size is decreased from 98 to 560 μm. When the sintering temperature is increased, foam thickness and strut thickness are decreased from 9.85 to 8.13mm and from 366 to 292 μm.

Keywords: electrostatic powder coating, Hastelloy, porous metal, pore properties, powder coating amount

1. Introduction

Porous metal is a metal having a lot of pores inside can be defined. The study of porous metal started in 1943 by B. Sosnik. That method is adding mercury to the molten aluminum to generate bubbles [1]. The porous metal has many excellent properties, such as function due to increase in specific surface area (heat exchange function, adsorption of other substances), reduction of relative density (light weight, soundproofing, shock absorption). Therefore, it is applied to various fields such as automobile industry, aviation, ship, railroad, sports and biomaterials [2]. The manufacturing method of the porous metal can be classified into a method using a molten metal and a method using a metal powder. Method using a molten metal includes melt gas injection, melt foaming and investment casting, etc [3-8]. Method using a metal powder include semi-solid foaming, gas expansion, MHS sintering, space holder technique, etc. [9-14]. Particularly, the powder porous metal can be manufactured by applying an alloy powder to a foam skeleton of a pure metal and performing a post-heat treatment process, and generally, it has a sponge-like three-dimensional network structure [15]. It is possible to manufacture various alloys of Fe, Ni and Cu type by using alloy powder, and it is possible to relatively easily and uniformly control the shape, size and distribution of pores. When such a powder porous metal is used as a filter material, it has been reported that the unburned solid-state particulate matter can be collected internally and the back pressure to the engine can be small [16]. Such a manufacturing method has the advantage of being able to reproduce with high reproducibility. However, this porous metal manufacturing process is based on pure metal foam, so it is subjected to complicated process and the price of pure metal foam is high. Also, since it is difficult to control the composition of the alloy, the material has a limit.

A way to overcome this limitation is electrostatic powder coating (ESPC) process. The metal powder is sprayed through the spray gun onto the sample. When the metal powder passes through the electric field formed between the corona electrodes of the spray gun tip charged with high voltage and the sample, the negative ion sticks to the metal powder. Then, it moves along the electric field formed at this time and is coated on the specimen. Metal powders of various compositions can be applied without limiting the material in the ESPC process, and when a foam-shaped template is used, it is possible to produce a porous metal body having a porosity of 80% or more. It is important to determine the optimal process conditions for each powder material because the process parameters such as the type of foam used as the template, the amount of coated metal powder and the sintering conditions influences the properties of the powder porous metal produced by this process.

Generally, Hastelloy haves excellent in corrosion resistance, chemical resistance, and widely used for chemical industry. In this study, in order to fabricating Hastelloy powder porous

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metal, Hastelloy powder uniformly coated PU (Polyurethane) foam directly by ESPC process, followed by the debinding and sintering to produce porous metal. Then it is investigated the influence on pore characteristics by controlling powder coating amount and sintering temperature.

2. Experimental

In this study, the ESPC process was used to produce Hastelloy powder porous metal. Figure 1 shows a flow chart of a process for manufacturing a porous metal. The powders used were Hastelloy powders prepared by gas atomization (PSI HERMIGA 100/25, UK Hot Gas Atomizer). For the gas atomization, a 2 kg charge of Hastelloy ingot (HVM. Co, Korea) was melted into a Magnesia crucible at temperature of 1600°C for 30 min under high purity argon atmosphere. After stabilization of melt temperature, the melt was ejected through the spray nozzle with the gas pressure of 50–60 bar. Upon gas atomization, the Hastelloy powders were collected and loaded onto a series of ASTM E11 standard sieves in order to obtain a specific particle size range, namely 10–45 μm. The powder was spherical as shown in Fig. 1 and had an average particle size of about 38.66 μm. Table 1 shows inductively coupled plasma (ICP) analysis of the component distribution of Hastelloy powder. Figure 2 shows a schematic diagram of the ESPC process for manufacturing a powder porous metal. In order to investigate the change of pore characteristics according to the amount of powder coating, ESPC were performed on PU foam (Jo – Spg. Co, Korea), which is 10 mm thickness and 2,000 μm cell size, by varying the powder coating amount to 3.0 kg and 3.5 kg. After powder coating is completed, finally apply binder and dry it to prevent desorption of powder at the time of transferring the test piece. The binder used was a mixture of BASF Lupasol stock solution and distilled water at a ratio of 1:9 (wt%). Based on the thermal analysis results of the binder and PU foam [17], the PU foam and the binder were completely debinded by heating to 1000°C at 5°C/min in an Ar gas atmosphere for 1 hour. The debinded test piece was sintered for 2 hours by changing the temperature to 1200°C~1300°C in a vacuum atmosphere (10⁻⁶ torr) to produce a Hastelloy powder porous metal. The sintering temperature was set to a temperature slightly under the melting range, which is about 1323°C~1371°C [18]. A scanning electron microscope (JSM - 5800, JEOL, Japan) was used to observe the size, shape and distribution of the pores of the manufactured Hastelloy powder porous metal. In addition, density and porosity measured using the Archimedes method.

3. Results and discussion

Table 2 shows the porosity, density, and thickness of the Hastelloy powder porous metal prepared through the ESPC process. The Hastelloy powder porous metal applied to a reactor for hydrogen production requiring a high reaction area and a low back pressure requires a high porosity of 50% or more [19]. In the case of the Hastelloy powder porous metal produced in this study, it confirmed that it shows porosity of 90% or more. As the Hastelloy powder coating amount increases, the porosity decreases at the same sintering temperature. As the sintering temperature increased, it decreased from 96.4% to 95.3% at 3.0 kg/m² and decreased from 95.6% to 94.4% at 3.5 kg/m². As the powder coating amount increases, the PU foam strut is coated with the powder thicker, and in some areas, the pore

<table>
<thead>
<tr>
<th>Composition</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>Mn</th>
<th>Si</th>
<th>V</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt. [%]</td>
<td>15.6</td>
<td>15.4</td>
<td>3.26</td>
<td>0.48</td>
<td>0.07</td>
<td>0.11</td>
<td>Balanced</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Powder : Hastelloy ( \left( D_{90} : 38.66 \mu m \right) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating on 10 mm thickness PU foam-ESPC ( \left( 3.0-3.5 \text{ kg/m}^2 \right) )</td>
</tr>
<tr>
<td>De - binding ( \left( \text{Ar gas } 1000^\circ C, 5^\circ C/\text{min} \right) )</td>
</tr>
<tr>
<td>Sintering - high vacuum ( 10^{-6} \text{torr}, 1200^\circ C-1300^\circ C, 5^\circ C/\text{min}, 2h )</td>
</tr>
</tbody>
</table>

Fig. 1. Flow chart of manufacturing process for Hastelloy powder porous metal

<table>
<thead>
<tr>
<th>Chemical compositions of Hastelloy powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>TABLE 1</td>
</tr>
</tbody>
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<table>
<thead>
<tr>
<th>Area density ( (\text{kg/m}^2) )</th>
<th>( 3.0 )</th>
<th>( 3.5 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sintering temperature</td>
<td>( 1200^\circ C )</td>
<td>( 1250^\circ C )</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>96.4</td>
<td>95.8</td>
</tr>
<tr>
<td>Foam density ( (\text{g/cm}^3) )</td>
<td>0.323</td>
<td>0.377</td>
</tr>
<tr>
<td>Thickness ( (\text{mm}) )</td>
<td>9.68</td>
<td>9.16</td>
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</tbody>
</table>

Fig. 2. Scheme of electrostatic powder coating process

<table>
<thead>
<tr>
<th>Properties of the Hastelloy powder porous metal</th>
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<tbody>
<tr>
<td>TABLE 2</td>
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clogging phenomenon blocking the foam cell structure also occurs, and at the same time, it can be inferred that the total porosity decreases due to the effect of high shrinkage occurring during the debinding and sintering process. The density of the manufactured Hastelloy powder porous metal increased from 0.323 g/cm³ to 0.497 g/cm³ as the powder coating amount and the sintering temperature increased. As the sintering temperature increases, it can be confirmed that from 0.323 g/cm³ to 0.419 g/cm³ at 3.0 kg/m² and from 0.391 g/cm³ to 0.497 g/cm³ at 3.5 kg/m². And it can be confirmed that the thickness of the manufactured Hastelloy powder porous metal increases as the powder coating amount increases at the same sintering temperature and decreases as the sintering temperature increases. As can be seen from the above, it is judged that the effect of the high shrinkage ratio occurred during the debinding and sintering process.

Figure 3 shows the results of the EDS mapping analysis of Ni, Cr, Mo and Fe elements (1300°C sintering conditions) and the photograph of the surface microstructures of the manufactured Hastelloy powder porous metal. From the results of the element mapping of the Hastelloy powder porous metal, it can be confirmed that elements are generated uniformly as a whole regardless of powder coating amount. Also, it can be confirmed that as the amount of the powder coating increases, the elements are more densely coated. From the results of the SEM of Hastelloy powder porous metal, the Hastelloy powders still maintain a rounded shape in the Hastelloy powder porous metal made at a low sintering temperature (1200°C). However, as the sintering temperature increases, the Hastelloy powders are bonded to the strut and show a smooth surface morphology.

Figure 4 shows the change in the pore size and the strut thickness depending on the powder coating amount and the sintering temperature of the manufactured Hastelloy powder porous metal. In the case of pore size, the diameter of pore contained in the unit cell measured, and the strut thickness measured based on the center of the strut. Figure 4(a) shows that as the sintering temperature increases, the pore size decreases to 978, 873, and 646 μm at the powder coated amount of 3.0 kg/m², and also decreases to 905, 753, and 560 μm at the powder coated amount of 3.5 kg/m². As shown in Fig. 4(b), the strut thickness decreases to 353, 342 and 292 μm as the sintering temperature increased at the powder coated amount of 3.0 kg/m², and also decreases to 366, 355 and 306 μm as the sintering temperature increased at the powder coated amount of 3.5 kg/m². As the amount of powder coating increased, the decrease of pore size and the increase of strut thickness resulted from the increase of the amount of powder coated on PU foam, which is similar to the decrease of porosity and increase of density. As the sintering temperature increases, the decrease in pore size and strut thickness is judged because shrinkage during sintering was accelerated.

Fig. 3. EDS and SEM images of Hastelloy powder porous metal with different powder coating amount and sintering temperature
4. Conclusions

In this study, the ESPC process was used to produce Hastelloy powder porous metal, and the pore characteristics were analyzed with the powder coating amount and sintering temperature. As the Hastelloy powder coating amount increased, the pore size decreased and the strut thickness increased. Also, as the sintering temperature increased, the pore size decreased and the strut thickness decreased. Based on the analysis result of EDS mapping, it confirmed that Hastelloy powder uniformly coated on the whole specimen. In this study, Hastelloy powder porous metal with various pore size and strut thickness were manufactured by controlling the powder coating amount and sintering temperature among various control factors of the ESPC process. In the future, we will investigate Hastelloy powder porous metal with various pore structures by changing the type of template and debinding conditions, and evaluate the physical properties according to the pore structure.

Acknowledgments

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