An Effect of Addition of NiO Powder on Pore Formation in Lotus-Type Porous Nickel

H. Onishi*1, S. Ueno*2 and H. Nakajima

The Institute of Scientific and Industrial Research, Osaka University, Ibaraki 567-0047, Japan

Lotus-type porous nickel with cylindrical pores was fabricated by unidirectional solidification in a mixture gas of hydrogen and argon. The pore size is significantly affected by the addition of NiO powder. The pore size decreases and the number density of the pores increases by the addition of NiO. It is concluded that NiO powder can serve as nucleation sites for the pore formation in the process of the solidification. [doi:10.2320/matertrans.MRA2008150]

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1. Introduction

Porous metals with cylindrical pores that are so-called lotus-type porous metals can be fabricated by unidirectional solidification using gas solubility gap between solid and liquid phases at their melting points.1,2) Since the specific strength of lotus-type porous metals does not change with the porosity when the loading direction is parallel to the pore direction, lotus-type porous metals are expected to be used for light-weight structural materials.3) The pores are formed at solid/liquid interface during the solidification. Since the pressure of inner pore is balanced with total atmospheric pressure during the solidification, the total volume of pores is inversely proportional to the total pressure according to Boyle’s law.2) Thus, the porosity of lotus-type porous metals can be controlled by total pressure. On the other hand, the pore size is significantly affected by the solidification velocity.4–6) The relations of the porosity and pore size of lotus-type porous metals with total pressure and hydrogen partial pressure are summarized in reference.2) The pore formation mechanism of lotus-type porous copper was discussed by Yamamura et al.7) In their report, the dissolved hydrogen atoms in liquid phase diffuse into pores at solid/liquid interface and the pores grow along solidification direction during the unidirectional solidification.7) The pore formation mechanism proposed by Yamamura et al. suggests that the solidification rate is a factor for controlling pore size. Actually, the pore size of lotus-type porous metals decreases with increasing solidification rate.4,6)

Suematsu et al. proposed a fabrication method of lotus-type porous nickel using moisture during the solidification in argon atmosphere.8) The pore size of the sample fabricated by this method is smaller than that of the sample fabricated in pressurized hydrogen atmosphere.8) Such smaller pore formation is attributed to formation of nucleation sites of the oxide. According to the following reaction (1), the moisture decomposes into hydrogen and metal oxide. The former produces hydrogen pores, while the latter may serve as the nucleation sites for the pores. Although the moisture could be produced by the reverse reaction (1), the moisture (H2O) itself cannot be dissolved into the molten nickel. Therefore, it is considered that the moisture does not contribute to evolve any pores in the solidified nickel. The experimental results of Suematsu et al.8) suggests that the pore size in lotus-type porous nickel can be controlled by the amount and/or particle size of NiO powder.

In this paper, the effect of NiO powder on the formation of pores was examined in order to elucidate the relation among pore size in lotus-type porous nickel, the amount and particle size of NiO powder.

2. Experimental Procedures

The solidification was conducted by mold casting under pressurized mixture gas of argon and hydrogen. 120 g of nickel ingot with 99.9% purity was heated in alumina crucible by induction coil and the molten nickel was poured into the mold whose bottom was made of copper and the side was made by molybdenum foil. The bottom of the mold was cooled by water chiller. An appropriate amount of NiO powder was put on the copper mold before the casting. To avoid the effect of moisture reported in the reference,8) the mold lubricant was not used for this experiment. The solidified sample was cut perpendicular and parallel to the solidification direction by wire-electric discharge machine.

The transversal and longitudinal cross sections of the samples were observed by optical and scanning electron microscope. The porosity and average pore diameter of the samples were measured by the image analysis (Win Roof, Mitani Corp.) of transversal cross section views at 5 mm height from the bottom.

3. Results and Discussion

Figure 1(a) and (b) show the longitudinal and transversal cross section views of the sample fabricated in the mixture gas of 0.65 MPa Ar and 0.15 MPa H2 using NiO powder whose weight is 0.5 g and particle size is 7 μm. These images

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1Graduate Student, Osaka University. Present address: Sumitomo Metals Industries Ltd., Osaka 541-0041, Japan
2Corresponding author, E-mail: ueno23@sanken.osaka-u.ac.jp
were obtained from the cross-sectional view at 5 mm height from the bottom of the samples. On the other hand, Fig. 1(c) and (d) show longitudinal and transversal cross section views of the sample fabricated under the same atmosphere without NiO powder. The porosity and average pore diameter calculated from Fig. 1 are 29% and 31 µm for (b) and 62% and 427 µm for (d), respectively. Apparently, the average pore diameter for (b) is smaller than that of (d). On the other hand, the aspect ratio of pores for (a), 2.2, is smaller than that of (c), 4.7. When the melt is solidified, supersaturated hydrogen in the solid diffuses into the pores. If the number density of pores increases, the amount of hydrogen which diffuses into each pore from the surrounding region decreases so that the pores cannot grow continuously in the direction of the solidification. Thus, the aspect ratio of the pores in the case of addition of NiO is shorter than that without NiO. Since the pore size decreases and the number density of pores increases by addition of NiO, it is considered that the oxide powders act as nucleation sites for pores.

Figure 2(a) and (b) show longitudinal cross section views of the sample fabricated in the mixture gas of 0.65 MPa Ar and 0.15 MPa H₂. During the melting, 0.1 g (a) and 0.3 g (b) of NiO powder with the particle size of 0.01 µm in particle size were added. The pore length in the case of addition of 0.1 g of NiO is much longer than that of 0.3 g. Since the number of nucleation sites for pore formation in the sample (b) is larger than that of (a) by the excess addition of NiO, the probability of pore nucleation for (b) is higher than that of (a). The supercooled and supersaturated hydrogen in the solid region in the vicinity solid/liquid interface may diffuse into pores. When the...
number density of pores is high, the hydrogen which diffuses to assist the growth of pores is not enough. Thus, the pores become shorter.

Figure 3 shows the porosity change with the amount of NiO powder. All samples in Fig. 3 were fabricated in the mixture gas of 0.85 MPa Ar and 0.15 MPa H$_2$. The porosity increases with increasing amount of NiO powder. It was suggested that about half of the hydrogen amount with the solubility gap between solid and liquid is released to the atmosphere not to contribute to the pore formation. However, if the number of the pore nucleation sites increases by addition of excess NiO powder, more insoluble hydrogen may be trapped by the nucleation sites. Thus, higher porosity was observed with increasing mass of NiO powder, as shown in Fig. 3. However, no significant effect of the particle size of NiO powder on the porosity was found.

Figure 4 shows the average pore diameter of the samples as a function of the mass of NiO powder. The pore diameter decreases with decreasing particle size of NiO powder. Since the number density of particles significantly increases with decreasing particle size when the same mass of NiO powder is added, the number of pores increases and the resulting pore size decreases as shown in Fig. 4. This may be attributed to the increase in the number of hydrogen atoms for formation of the pores by NiO powder addition. On the other hand, the average pore diameter monotonously increases with increasing amount of NiO powder.

4. Conclusion

Addition of NiO powders is effective to produce small pores in lotus-type porous nickel. The amount of NiO powder and its particle size affect the porosity, pore diameter and pore morphology (aspect ratio) in lotus-type porous nickel. The pore diameter significantly depends on the NiO particle size. Thus, NiO powder may serve as nucleation sites for the hydrogen pore formation in lotus-type porous nickel.

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