Fabrication of lotus-type porous iron and its mechanical properties

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Abstract

Porous iron whose long cylindrical pores are aligned in one direction has been fabricated by unidirectional solidification of the melt in a mixture gas of hydrogen (nitrogen) and argon. Both hydrogen and nitrogen saturated in the molten iron are rejected at the solid–liquid interface during the solidification due to the difference of solubility between the liquid and the solid. The gas pores are evolved from the hydrogen (nitrogen) insoluble in the solid iron, which grow unidirectionally. The porosity is controlled by the partial pressures of hydrogen (nitrogen) and argon during melting and solidification. By increasing the partial pressure of argon gas the pore formation is suppressed, since the pressure and, therefore, the density of the hydrogen (nitrogen) gas in the growing pore are increased with the total pressure of the atmosphere.

The nitrogen concentration in solid iron fabricated under nitrogen atmosphere increases linearly with partial pressure of nitrogen, leading to the improvement of mechanical properties of the porous iron. The ultimate tensile strength and the yield strength of the porous iron with the pore orientation parallel and perpendicular to the tensile direction are about twice as high as those under hydrogen atmosphere. Such superior strength is attributed to the solid-solution hardening due to solute nitrogen atoms in iron matrix.

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1. Introduction

There has been a great interest in fabrication of porous metals as it opens a path to the development of lightweight structural materials with thermal, acoustic and energy absorption characteristics [1,2]. Recently, a new type of porous metal whose long cylindrical pores are aligned in one direction has been fabricated by unidirectional solidification method in the hydrogen atmosphere under high pressure [3–15]. These porous metals should be distinguished from the conventional porous metals whose pores are almost isotropic. Hereafter, we designate these porous metals with elongated pores lotus-type porous metals, because they look like lotus roots. The tensile properties of lotus-type porous copper whose pores are aligned in one direction are superior to those of conventional porous metals [14]. Thus, such long pore-aligned porous metals are expected as a new category of engineering materials.

Furthermore, it is well known that the temperature dependence of nitrogen solubility in solid and liquid of iron is similar to that of hydrogen [16]. It is noticed that an invariant reaction of ‘gas-evolution crystallization reaction’ [9] takes place in Fe–N system, in which the iron melt dissolving nitrogen is solidified to transform into the primary solid solution and nitrogen gas phase. Therefore, utilizing the solubility difference of nitrogen in the liquid and solid states, it is possible to fabricate porous iron using the nitrogen. In the present study, the lotus-type porous iron was fabricated in hydrogen atmosphere and in nitrogen atmosphere, and the effect of porosity and pore size on the atmospheric pressure during fabrication was investigated. Furthermore, the tensile properties of lotus-type porous iron were also investigated.

2. Experimental procedure

The fabrication apparatus of the porous iron consists of an alumina crucible (40 mm external diameter, 30 mm internal diameter, and 100 mm length) surrounded by an induction heating coil and a mould with water-cooled copper plate as shown in Fig. 1. These are installed in a high-pressure chamber. High purity iron (99.99% Fe) was
melted in the crucible in a vacuum, and then high-pressure mixture gas of hydrogen (nitrogen) and argon was introduced into the chamber. The pressure was measured by a pressure gauge sensor and the partial pressures of hydrogen (nitrogen) and argon were controlled in the range from 0 to 2.8 MPa. The temperature of the melt in the crucible was monitored by an infrared pyrometer (Model IR-AP, Chino Co.). The melting temperature was set to be 1973 K. The crucible was rotated by 90° to pour the molten iron into the mould whose bottom plate was water-cooled. Thus, the molten iron was solidified unidirectionally upwards. The ingots obtained were 35 mm in diameter and maximum 80 mm in height, and contained various levels of porosity depending on the partial pressures of gases. The nitrogen concentration of porous iron was analyzed by means of Oxygen–Nitrogen Determinator (Model TC300, LECO Co.). The porosity is calculated from the following expression:

\[
\text{Porosity} = 1 - \frac{\text{Apparent density of porous iron}}{\text{Density of non-porous iron}}
\]

The apparent density of the individual specimens was calculated from measurement of its weights at air and in the water through Archimedes’ method.

The ingots were cut using a spark-erosion wire cutting machine (Model A320D, Sodick Co.) to prepare specimens for tensile tests so that the tensile directions were parallel and perpendicular to the pore growth direction. The gage length and the gage section of the specimens were 12.5 mm and 1 x 8 mm², respectively. Uniaxial tensile tests were performed at room temperature with a Universal Testing Machine (Model 4482, Instron Co. Ltd). The strain rate was set up to be 10⁻² s⁻¹. The strain was measured by using an extensometer. The ultimate tensile strength was evaluated from the stress–strain curve.

3. Results and discussion

3.1. Lotus-type porous iron fabricated in hydrogen atmosphere

Fig. 2 shows the typical optical micrograph of the parallel and the perpendicular cross-sections of lotus-type porous iron fabricated under the total pressure 2.5 MPa (0.5 MPa H₂ and 2.0 MPa Ar). Fig. 3 shows the dependence of porosity of lotus-type porous iron on the partial pressures of hydrogen and argon during the melting and solidification processes. Table 1 shows the condition of atmospheric pressure, the measured porosity and the pore size. Little pores were formed when the pressures of hydrogen...
and argon were 0.1 and 1.7 MPa, and 0.3 and 2.5 MPa, respectively. At a given hydrogen pressure, the porosity decreases with increasing argon pressure. At a given argon pressure, the porosity increases linearly with hydrogen pressure.

Assuming that the growth rate during solidification is constant, the volume (porosity), $V$, of gas pores formed during solidification can be explained on simple mechanism, as illustrated by the well-known general gas law assuming that the gas in pore behaves as perfect gas [17]:

$$V = \frac{nRT}{P},$$  \hspace{1cm} (2)

where $P$ is the applied atmospheric pressure, $R$ the gas constant 8.314 (J mol$^{-1}$ K$^{-1}$), and $n$ the amount of gas. The volume of the pores is directly proportional to the amount of gas, $n$, present in solution, and is inversely proportional to the applied pressure. The pressure dependence of porosity shown in Fig. 3 can be understood as follows. According to the Sievert’s law, $n$ increases with increasing partial pressure of hydrogen at a constant total atmospheric pressure so that the porosity increases. On the other hand, $P$ increases with increasing partial pressure of argon at a constant partial pressure of hydrogen and then the porosity turns to decrease.

### 3.2. Lotus-type porous iron fabricated in nitrogen atmosphere

Fig. 4 shows the typical optical micrographs of the cross section at the bottom part of the ingot of lotus-type porous iron fabricated under the pressure of 1.0 MPa N$_2$ and 0.5 MPa Ar. The overall porosity was measured as functions of the partial pressures of nitrogen and argon as shown in Fig. 5. The porosity was found to be affected by the nitrogen and argon pressures. At a given nitrogen pressure, the porosity decreases with increasing argon pressure. At a given argon pressure, the porosity increases linearly with nitrogen pressure. This tendency agrees with that of porous iron fabricated in hydrogen atmosphere as shown in Fig. 3.

On the other hand, when the total gas pressure of nitrogen and argon was kept constant at 2.0 MPa, the measured nitrogen concentration of the lotus-type porous iron depends on the partial pressure of nitrogen and ranges from 0.06 to 0.23.

### Table 1

Partial gas-pressure condition, porosity and pore size of lotus-type porous iron

| Hydrogen pressure (MPa) | Argon pressure (MPa) | Porosity (%) | Pore size (µm) |
|-------------------------|----------------------|--------------|----------------|
| 0.1                     | 1.7                  | 1.0          | –              |
| 0.1                     | 2.7                  | 0.0          | –              |
| 0.3                     | 2.5                  | 1.8          | 174 ± 115      |
| 0.5                     | 1.5                  | 53.0         | 420 ± 226      |
| 0.5                     | 1.8                  | 36.9         | 168 ± 74       |
| 0.5                     | 2.0                  | 36.5         | 250 ± 107      |
| 0.5                     | 2.3                  | 31.0         | 186 ± 96       |
| 0.8                     | 1.8                  | 44.7         | 241 ± 142      |
| 0.8                     | 2.0                  | 42.7         | 228 ± 132      |

Fig. 4. Optical micrographs of lotus-type porous iron fabricated in nitrogen atmosphere (1.0 MPa N$_2$ and 0.5 MPa Ar): cross section (a) perpendicular and (b) parallel to pore axis.
0.135 mass%. According to the solubility curve of nitrogen in iron [16], the nitrogen solubility in \(\gamma\)-iron decreases with increasing temperature and the maximum solubility is 0.135 mass% at the lowest temperature of the \(\gamma\)-phase, 1184 K. In \(\delta\)-iron the nitrogen solubility increases linearly with temperature and the maximum solubility is known as 0.06 mass% at the temperature just below the melting point of iron, 1809 K. Thus, it is reasonable that the measured nitrogen concentration range is in between these two limits of the solubility [15].

### 3.3. Tensile properties

Typical stress–strain curves of the lotus-type porous iron specimens fabricated by hydrogen or nitrogen gas are shown in Fig. 6. The tensile direction is parallel to the direction of the pore axes. The curves show a linear elastic behavior at small strains, followed by yield and strain hardening up to the peak stress. Drastic difference in the strength of the specimens fabricated with hydrogen and nitrogen was observed even at similar porosity; the ultimate tensile strength of the specimen fabricated by nitrogen is about twice higher than that by hydrogen. According to the chemical analysis, hydrogen of 27.7 mass ppm is contained in the specimens fabricated under hydrogen, which is not considered to affect the strength of iron, since the tensile data as shown in Fig. 7 extrapolated to the zero porosity are good fit to the result of pure non-porous iron which hardly contains hydrogen. On the other hand, nitrogen as much as 0.0873 mass% contains in the specimens fabricated with nitrogen atmosphere. Thus, such remarkable enhancement of the tensile strength is attributed to solid solution strengthening due to solute nitrogen atoms.

Both yield strength and ultimate tensile strength of specimens with cylindrical pore orientation parallel to the tensile direction are much higher than that perpendicular as shown in Fig. 7. Such anisotropic strength was interpreted by the previous work on lotus-type porous copper [14]. For the specimen with cylindrical pores aligned parallel to the tensile direction, the strength is expressed by

\[
\sigma = \sigma_0(1 - p),
\]

3.3. Tensile properties

![Fig. 5. Porosity dependence on the partial pressures of nitrogen and argon gas.](image1)

![Fig. 6. Stress–strain curves for lotus-type porous iron with 50.1 ± 2.6% porosity fabricated by (a) nitrogen or (b) hydrogen gas: the tensile direction is parallel to the direction of the pore axes.](image2)

![Fig. 7. Porosity dependence of (a) yield strength and (b) ultimate tensile strength of lotus-type porous iron fabricated with hydrogen and nitrogen in the direction parallel and perpendicular to pore growth orientation.](image3)
where $\sigma$ and $\sigma_0$ is the strength of porous and non-porous materials, respectively, and $p$ is the porosity. For the specimen with cylindrical pores oriented perpendicular to the tensile direction, the strength is given by

$$\sigma = \sigma_0(1 - p)^3.$$  \hfill (4)

It is found that the experimental results are in good agreement with the dotted lines (Fig. 7) estimated from these equations. This fact indicates that pores whose axes are aligned parallel to the tensile direction cause little stress concentration; the specific tensile strength is constant in spite of the pore existence in the porous iron. On the other hand, the ultimate tensile strength with the pore orientation perpendicular to the tensile direction can be quantitatively explained in terms of the stress concentration; the stress is accumulated in the vicinity of the pores.

4. Conclusion

The lotus-type porous iron having long aligned pores via melt-route process was fabricated in nitrogen atmosphere as well as hydrogen atmosphere. At a constant total pressure of the mixture gas, the porosity increases linearly with partial pressure of hydrogen (or nitrogen). At a given partial pressure of hydrogen (or nitrogen), the porosity decreases with increasing partial pressure of argon. The ultimate tensile strength and the yield strength of the porous iron fabricated in nitrogen with the pore orientation parallel and perpendicular to the tensile direction are about twice higher than those under hydrogen atmosphere. Such superior strength is attributed to the solid-solution hardening due to solute nitrogen atoms in iron matrix.

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