Fabrication of Porous Aluminum by Spacer Method Consisting of Spark Plasma Sintering and Sodium Chloride Dissolution

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Porous aluminum with a porosity of 78% and pore size of 850–1000 μm was fabricated under various sintering pressure, sintering time and raw Al powder size conditions by the spacer method consisting of spark plasma sintering (SPS) and sodium chloride (NaCl) dissolution. The effects of the fabrication conditions on compressive properties of the porous Al were investigated. The sintering pressure of 20 MPa and sintering time of 10 min were needed to fabricate robust porous Al under the sintering temperature of 843 K and raw Al powder size of 3 μm. Also, the porous Al specimen fabricated from Al powder of 300 μm exhibited much lower flow stress than those fabricated from Al powder of 3 and 20 μm when employing the temperature of 843 K, the pressure of 20 MPa and the duration time of 10 min. This indicates that the raw Al powder size is needed to be much smaller than the spacer size. This is because the Al particle cannot touch with adjacent Al particles when the Al powder size is comparable to the spacer size.

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1. Introduction

Porous aluminum is one of promising structural ecomaterials because of its ultralow density and excellent energy absorption capacity, etc.¹,² Its deformation under a nearly constant flow stress, called plateau stress, over a wide strain range is the reason why the porous Al is expected to be used as a shock absorber.³ In particular, the application of porous Al in vehicle industries is often proposed for its high potentials as an ultralight shock absorber.⁴ To date, various manufacturing ways of porous metals including porous Al have been developed,²⁻⁹ and some of them are now being applied to the commercial production of porous metals.²,⁴ The control of pore characteristics such as porosity, pore morphology and pore size distribution is one of problems to be solved in applying porous aluminum to shock absorbers of vehicles, because a high reliability is required to protect a human body from crush accidents.

The spacer method, which is one of the manufacturing methods of porous metals, can control the pore characteristics for following reasons.⁰⁻¹² (1) Adjustment of mixing ratio of spacer and metal powder results in porous metals with finely controlled porosity. (2) Preparation of sieved spacer particles can narrow the pore size distribution. Recently, spark plasma sintering (SPS) and dissolution of spacing NaCl particles have been combined for efficient production of porous Al.¹³ In the previous work,¹⁴ the effects of the sintering temperature on compressive properties of porous Al fabricated by SPS and NaCl dissolution have been investigated. In the present paper, the effects of other conditions such as the sintering pressure, sintering time and raw Al powder size on the compressive properties of porous Al are investigated.

2. Experimental Procedure

Commercially available 99.9% pure aluminum powder with average diameters of 3, 20, or 300 μm and sieved NaCl particles with the sizes of 850–1000 μm were selected as raw materials. They were thoroughly mixed in a volume ratio of 22:78. The mixture was then subjected to the spark plasma sintering equipment (SPS-515S by Sumitomo Coal Mining Co., Ltd.). In this work, the sintering temperature (= the temperature of the graphite die wall) was fixed to 843 K. The sintering pressure and sintering time (defined as the duration time during which the sintering temperature was kept to 843 K) were varied. Subsequently, the cylindrical porous Al was obtained by processing the sintered compacts with water washing to remove space-holding NaCl particles. As a reference, bulky Al specimens, which represent the similar mechanical properties with those of the cell wall materials of the porous Al, were also fabricated under the same sintering conditions as those used for the porous Al.

Compressive tests on the porous Al specimens were carried out at room temperature. The dimensions of the porous Al specimens were 20 mm in diameter and 22 mm in length. In addition, compressive and tensile tests on the bulky Al specimens were performed. Their dimensions were 20 mm in diameter and 22 mm in length for the compressive tests, and 8 mm in gauge diameter and 10 mm in gauge length for the tensile tests. The crosshead velocity was 1 mm/min for all compressive and tensile tests. The cross sections of the specimens were prepared by grinding and polishing, and observed with an optical microscope and a scanning electron microscope (SEM).

3. Results and Discussion

3.1 Effect of Sintering Pressure

Porous Al specimens were fabricated at various sintering
pressures of 8–24 MPa, where the sintering temperature and time were fixed to 843 K and 10 min, respectively. The average particle size of raw Al powder was 3 µm. Figure 1 shows the cross sections of the porous Al specimens fabricated at the sintering pressures of 20 and 8 MPa. It appears that the pore sizes were in agreement with the sizes of space-holding NaCl particles of 850–1000 µm. The mass and dimension measurements proved that the porosity of the fabricated porous Al was approximately 78% for each specimen, indicating that all NaCl particles were successfully removed. There were no significant differences in pore characteristics between the specimens fabricated at various sintering pressures of 8–24 MPa.

Figure 2 shows the results of compressive tests on the porous Al specimens fabricated at various sintering pressures. The stress-strain curves for the porous Al specimens fabricated at 12 MPa or more exhibited three stages; (1) the elastic region at an initial stage of deformation, (2) the plateau region with a nearly constant flow stress of about 5 MPa over a wide range of strain and (3) the densification region with a steep increase of the flow stress at higher strains. These three regions mean the compressive stress-strain curves for porous metals. However, the specimen fabricated at 8 MPa showed a lower flow stress, and did not show the densification region clearly because of its brittle fracture.

Inspection of Fig. 2 revealed that the flow stresses in the densification regions of the porous Al specimens fabricated at 12 and 16 MPa were lower than those at 20 and 24 MPa. This is because the local fragmentation of the formers occurred during their deformation. Therefore, the sintering pressure of at least 20 MPa is required to fabricate robust porous Al by this spacer method.

Cell walls of the deformed porous Al specimens fabricated at the sintering pressures of 20 and 8 MPa are shown in Fig. 3. It should be noted that the cell walls of the porous Al
specimen fabricated at 20 MPa were bent without fracture; however, the cell walls of the porous Al specimen fabricated at 8 MPa underwent brittle fracture.

Figure 4 shows microstructures of the cell walls of the porous Al specimens fabricated at 20 and 8 MPa prior to the compressive tests. No voids were found in the cell walls of the former, while some voids were observed in those of the latter. These voids may be generated by the insufficient connection between Al particles caused by the lower sintering pressure.

Figure 5 shows the results of compressive and tensile tests on the bulky Al fabricated at the sintering pressures of 20 and 8 MPa, where the sintering temperature of the bulky Al specimens was fixed to 10 min and the raw Al powder size was 3 μm. In compression, both of the bulky Al showed good ductility; although the flow stress of the bulky specimen fabricated at 8 MPa was lower than that of the bulky specimen fabricated at 20 MPa. However, in the tension test result, the bulky specimen fabricated at 8 MPa showed an elongation of 2%. It is much lower than that in fabricating at 20 MPa. Therefore, the bulky Al fabricated at 8 MPa was very brittle in tension, compared to in compression.

When a porous material is compressed, the cell walls are bent or buckled, and tensile stresses as well as compressive stresses are locally generated at the cell walls. Markaki and Clyne reported that the ductility of cell walls of porous aluminum is important especially when local tensile stresses occur in the cell walls. In the present investigation, when a porous Al specimen fabricated at a low sintering pressure of 8 MPa was compressed, the brittle cell walls brittle in tension were fractured with no sufficient load transfer, and resulted in a lower flow stress. Therefore, the ductility of cell walls in tension is important to evaluate the compressive properties of the porous Al.

3.2 Effect of Sintering Time

Porous Al specimens were fabricated at various sintering times under the sintering temperature of 843 K, the sintering pressure of 20 MPa and the raw Al powder size of 3 μm. Observation of their cross sections showed no significant difference in pore characteristics between the porous Al fabricated at various sintering times. This is the same trend as derived in the previous section. Figure 6 shows the nominal stress-nominal strain curves for the porous Al specimens fabricated at various sintering times, where the sintering temperature and the sintering pressure were 843 K and 20 MPa, respectively.
plateau regions with the nearly constant flow stress of about 5 MPa. The flow stress for the porous Al specimens sintered for 3 min was significantly varied during its deformation. Also, the compressive stress in the densification region of the specimen with the sintering time of 6 min was lower than those of the specimens with longer sintering times. This specimen was fractured partially during deformation. These undesirable results for the porous Al with the shorter sintering times are probably due to the poor bonding between Al powder particles. The cell walls of the porous Al specimens sintered for a shorter time may be brittle in tension, in the same way as discussed in the previous section. Therefore, it is concluded that the sintering time enough to fabricate robust porous Al is 10 min under the sintering conditions investigated above.

3.3 Effect of Raw Al Powder Size

Porous Al specimens were fabricated from Al powder with different average sizes, where the sintering temperature, the sintering pressure and the sintering time were 843 K, 20 MPa and 10 min, respectively. The results of their compressive tests are shown in Fig. 7. The porous Al specimens fabricated from Al powder of 3 and 20µm showed the plateau region in the stress-strain curves, and their plateau stresses were 5.0 and 3.8 MPa, respectively. However, the porous Al specimen fabricated from coarse raw Al powder of 300µm exhibited the very low flow stress of nearly zero without the elastic, the plateau and the densification regions.

Figure 8 shows the results of compressive and tensile tests on the bulky reference Al specimens fabricated from Al powder with the average sizes of 3 and 300µm, where the sintering temperature, sintering pressure and sintering time were 843 K, 20 MPa and 10 min, respectively. These sintering conditions are the same as those of the porous Al. The bulky Al specimens fabricated from Al powder with the average size of 300µm showed a good ductility both in compression and in tension, although its flow stresses were lower than those of the bulky Al specimen in using 3µm Al powder. It should be noted that the porous Al fabricated from the coarse Al powder of 300µm showed the very low flow stress of nearly zero, as shown in Fig. 7, although the ductility in tension of the bulky Al specimen was not poor even in case of the same coarse powder. That is, the Al powder with the size of 300µm was sufficiently sintered. However, in fabrication of porous Al from Al powder with the size of 300µm, as illustrated in Fig. 9, Al powder particles cannot touch with adjacent Al particles when mixed with NaCl particles, because the size of the spacing NaCl particles is of the same order as the size of Al particles, and the volume fraction of NaCl particles is quite large. In other words, the spacing NaCl particles geometrically prevent Al particles from touching and metallurgically bonding each other. In such a case, mechanical properties are poor for even the porous Al fabricated under the suitable sintering temperature, pressure and time. Therefore, the
particle size of Al powder size should be much smaller than that of the NaCl particles to fabricate robust porous Al by the spacer method.

4. Conclusions

Effects of sintering pressure, sintering time and raw Al powder size on compressive properties of porous Al fabricated by the spacer method consisting of SPS and NaCl dissolution were investigated. The conclusions are remarked as follows.

(1) The sintering pressure of 20 MPa and the sintering time of 10 min were needed to fabricate the porous Al showing the deformation characteristics of the elastic region, the plateau region and the densification region in the compressive stress–strain curves, when employing the sintering temperature of 843 K and the raw Al powder size of 3 μm.

(2) When a porous Al specimen fabricated at the low sintering pressure of 8 MPa was compressed, the brittle cell walls in tension were fractured with no sufficient load transfer, and resulted in the lower flow stress. Therefore, the compressive properties of the porous Al strongly depend on the ductility of cell walls in tension.

(3) The porous Al specimens fabricated from Al powder of 3 and 20 μm showed the plateau region in the stress-strain curves, and their plateau stresses were 5.0 and 3.8 MPa respectively, under the sintering temperature of 843 K, the sintering pressure of 20 MPa and the sintering time of 10 min. However, the porous Al specimen fabricated from raw Al powder of 300 μm exhibited a very low flow stress of nearly zero due to its poor bonding between raw Al powder particles. Accordingly, the particle size of Al powder should be much smaller than that of NaCl particles to fabricate robust porous Al specimens.

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