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Metallic cellular structure fabricated by pulse current assistant hot quasi-isostatic pressing

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During a pulse-current hot pressing (PCHP) by using a spark-plasma-sintering (SPS) system, the pressure applied on the particles in the die is axis-dominated. Therefore, an inhomogeneous deformation of the particles, especially soft particles, occurs in the sintering process. In this report, a novel method, pulse-current hot quasi-isostatic pressing (PCHIP), was introduced for fabrication of metallic cellular structures with a very fine cellular structure (cell size 10-30μm). Polymer-solid spheres coated by Ni-P alloy layer was used in this study. After the sintering by PCHIP method by using the SPS system, the metallic layers of the particles were joined together; and then the metallic cellular structure was obtained. Microstructures and mechanical properties of the material were compared with those of the materials fabricated by PCHP.

1. INTRODUCTION

Cellular metals are well known to have many interesting combinations of mechanical, physical and chemical properties. The scale of the cell size produced by normal methods is in range of 0.1mm to several centimeters [1]. The techniques for finer cellular structure are lacking except for the sintering method.

Hollow sphere made by Ni, Cu and steel can be used to fabricate cellular structure by bonding, brazing or sintering [2],[3]. In these methods, the spheres can even be assembled in a special lattice structure. Although the size of the hollow spheres is quite controllable, the scales of the cellular structures fabricated by the mentioned references were rather large.

A novel metallic material with a very fine cellular structure (cell size: in level of a few decades of microns) has been developed by sintering alloy-coated polymer-solid spheres using normal sintering [4,5]. After the sintering, though the metallic coated layer jointed together, and the polymer materials inside the metallic cellular structure were consumed or carbonized for the high temperature .

Figure 1 shows the process of the fabrication of the metallic cellular structures by the normal sintering method [4]. However, the material inside the cell was thought to benefit the mechanical properties of the cellular structure, such as damping property and stiffness. So, the rapid sintering technique is required. Therefore, pulse current hot pressing (PCHP) method using spark plasma sintering (SPS) system has been developed to fabricate the cellular metal with the polymer remaining by using the alloy-coated polymer-solid spheres [6].

In this report, a new method, pulse-current hot quasi-isostatic pressing (PCHIP) sintering [7] was introduced for the fabrication of the metallic cellular structure. The microstructures and mechanical properties of the materials fabricated by PCHP and PCHIP were compared.

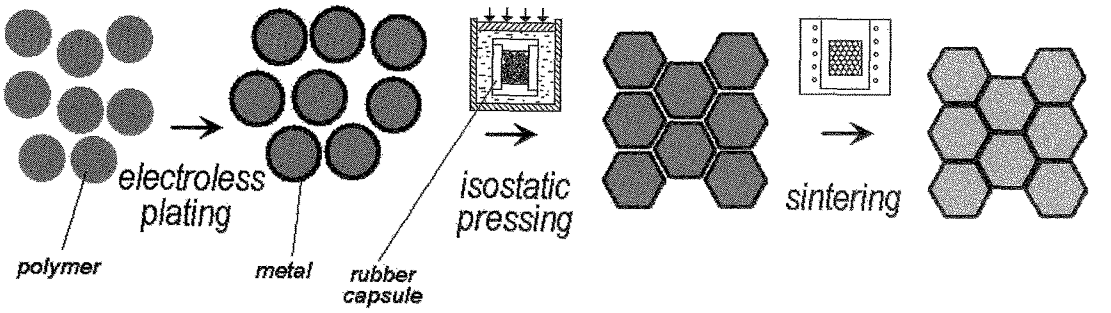

Figure 1 *Fabrication process of metallic cellular structure by former sintering method [6].*

2. EXPERIMENTAL METHODS

2.1 Sintering methods

To perform PCHP and PCHIP, the SPS system was used. The SPS system was developed by K. Tokita mentioned in Ref. [8].

The SPS method is a type of solid compression sintering method, similar to the hot pressing (HP) but faster than the HP. During the SPS process, powder particles were filled in a die and pressed by a pair of graphite punches, pulse electric current flows through the particles, an efficient heating located at the contact area between the particles is offered [8]. If the current is large enough, plasma may generate between particles [9]. Nevertheless, the process of how and when the plasma will be generated has not been clarified. To avoid being entangled by the plasma, this technique is sometimes also called pulse-current hot pressing (PCHP) [10]. It is a next-generation technique to sinter at a lower temperature and in shorter actuation time compared with the normal HP.

In the PCHP process, the pressure applied on the particles in the die is axis-dominated. The pressure in direction of the pressing axis is larger than that normal to the pressing axis. Actually, there is a distribution of the pressure in the radial in the section of the particles compact. The distribution of the pressure always causes an inhomogeneous deformation of the particles especially those soft sphere particles in the sintering process. So the cellular structure fabricated with the SPS system is flattened to the pressure. The process of the PCHP method is shown in Figure 2. In order to improve the axis-dominated pressing condition in the PCHP method, a quasi-pressing method was developed. In this method, green compact pre-produced by cold isostatic pressing was used for hot pressing in a quasi-isostatic condition (Figure 3).

The process of the PCHIP method is as follows. (1) The polymer particles coated with Ni alloy were assembled in a flexible rubber capsule and the green compact was formed by a cold isostatic pressing method. (2) The green compact was placed in the graphite die surrounded by fine zirconia sphere particles. (3) Pulse current was applied on the green compact. The pulse current was generated by an SPS system (SPS-515S, Sumitomo Coal Mining Co., Ltd.) and passed through the green compact from the graphite powder and punch. (4) A high efficient heating was occurred. A cellular material can be obtained. Detail of this method will be mentioned in another paper [7].

2.2 Powders for specimens

The alloy-coated polymer-solid (phenol resin) sphere particles were fabricated by electroless plating as shown by first two steps in Figure 2 and Figure 3. The coating alloy is nickel-phosphorus (2.5wt%) and the thickness of the alloy is in range of 1 and 5μm depending on the diameter of the particle. The average thickness of the

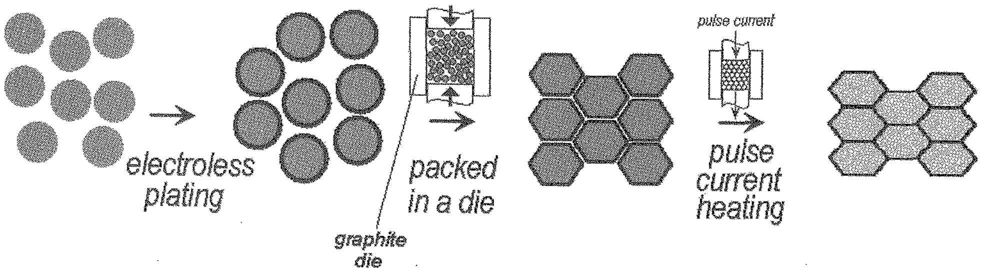

Figure 2 *Fabrication processes of metallic cellular structures by PCHP.*

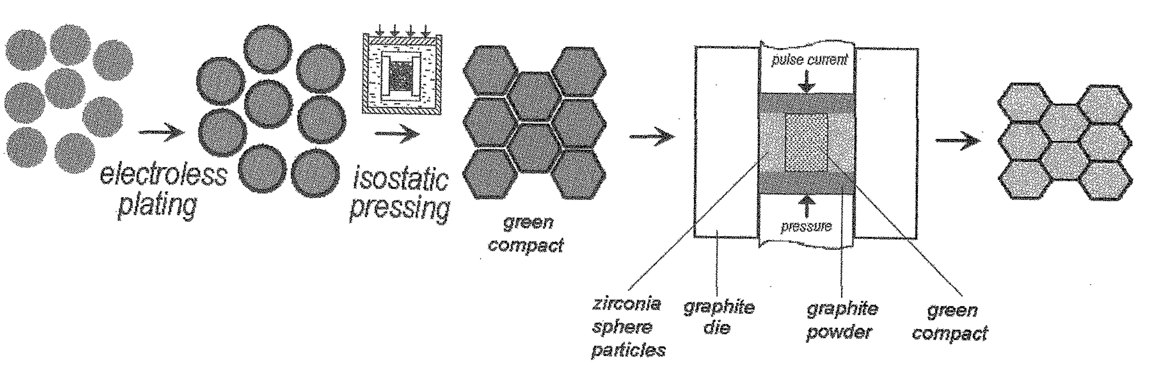

Figure 3 *Fabrication processes of metallic cellular structures by PCHIP sintering.*

coating alloy was about 3.5 μm. The diameter of the particle was distributed in a range between 5 and 120 μm. The size distribution of these spheres particles was shown in Figure 4.

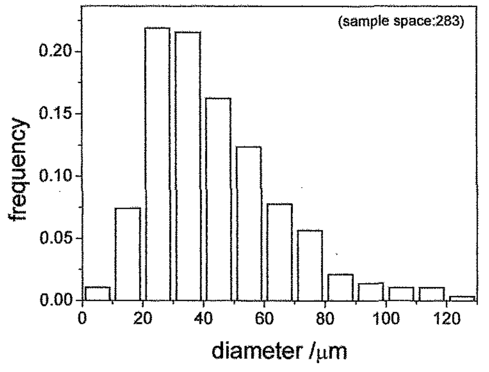

Figure 4 *The size distribution of the phenol resin spheres coated with Ni-P.*

Assuming that all particles were deformed into polyhedral shape after sintering (which means that there is no gaps among the cells and there is no change of the volume of the particle after sintering), the theoretical volume percent of the alloy is about 37%.

2.3 Microstructure observation

The cross-sections of the specimens by PCHP and PCHIP were cut and published. For the observation, scanning electron microscope (SEM) was used.

2.4 Mechanical properties

To measure the mechanical properties, the internal friction and the Young's modulus were measured by a resonance vibration method (Nippon Techno-Pulse Co.). The measurements were performed from room temperature to 500°C.

3.RESULTS AND DISCUSSION

3.1 Microstructures

Figure 5 shows the cellular structures of the specimens fabricated by the PCHP method and the PCHIP method. Figure 6 shows a higher magnification of the same specimen shown in Figure 5 (b).

As seen in Figure 5 (a), it can be found that most of the cells were deformed by the pressure applied by the SPS system during the sintering process. It can also be found that most of the cells in Figure 5(b) are deformed in equiaxed. From the high magnification image of Figure 6, it can be seen that the adjacent spheres joined quite well and several spheres were deformed by the squeezing from the neighbors. By comparing with Figures 5(a) and 5(b), it is clear that PCHIP method provided the method for sintering the particles under an isostatic-pressing condition.

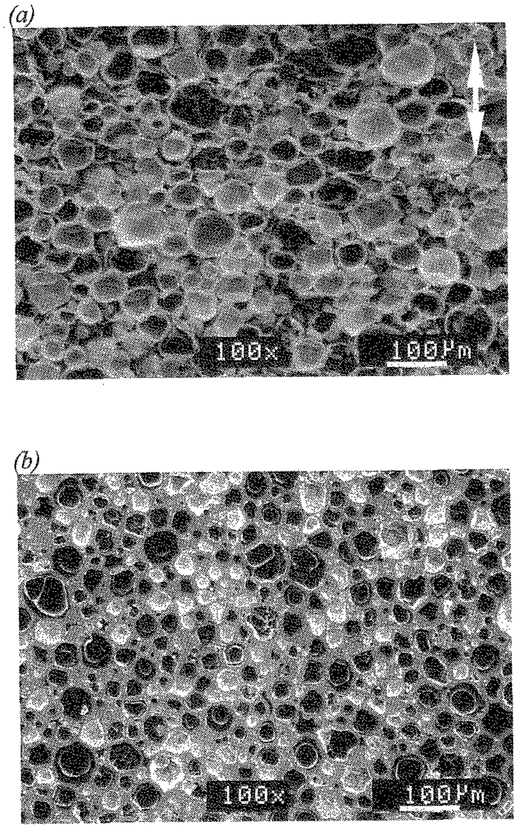

Figure 5 *SEM images of cellular materials fabricated by PCHP (a) and PCHIP (b). The arrow in (a) indicated the direction of current and loading pressure.*

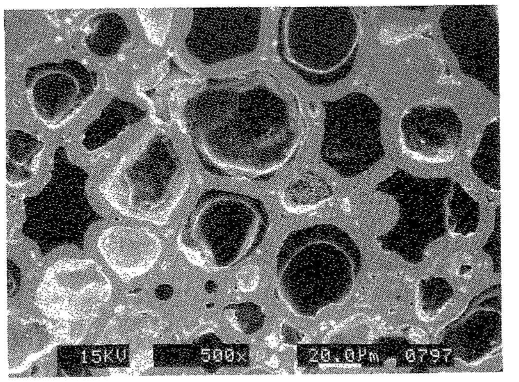

Figure 6 *Close-up image of the cellular structure fabricated by PCHIP method.*

Quantitative analysis was done by using an optical microscope. The area ratio of the metallic part in the cross-section of the sample fabricated by PCHP is about 41% and larger than the theoretical one (37%). Furthermore, the area ratio of the alloy in the cross-section of the sample fabricated by PCHIP is about 44%, larger than the theoretic one and also larger than the PCHP one. These results come from two reasons. One is the compression of the particles. After sintering the volume of space inner the alloy coats decreases while the volume of the alloy had no change. The other reason is the recessing of the large spheres by the squeeze from their neighbors.

Comparing with normal PCHP, the presented PCHIP method seems to have two advantages. One is the fewer amount of cracks in the sample by this method than that of PCHP. The other is the shape of the cells deformed in equiaxedly.

It should be thought that there is a temperature distribution along the radius direction. While the pulse current passed through the particles of the green compact during the sintering, the temperature at the joints reaches a very high value by the Joule heating. The temperature at the joints is important for the sintering process but not important for the polymer inside the cell.

3.2 Damping tests

Figure 7 shows internal friction and Young's modulus of Ni-P alloy cellular specimens fabricated with different process parameters as a function of temperature in acoustic frequencies (about 750-800 Hz) by using a resonance-vibration method.

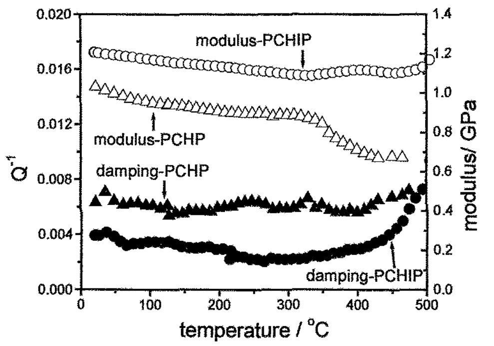

***Figure** 7 Internal friction and Young's modulus of specimens as functions of temperature.*

The internal friction of the specimen fabricated by PCHIP is lower than that by PCHP. When the temperature is higher than 450°C, the internal friction of the specimen fabricated by the PCHIP increased more than that by PCHP. For the specimen fabricated by PCHP method, Young's modulus of the specimen decreases with increasing temperature from 20 to about 400°C and it decreased immediately at about 350°C. It was thought that the specimens were softened when the temperature increased over 350°C. However, the Young's modulus of the specimen fabricated by the PCHIP method kept a constant value and then increased when the temperature reaches 470°C while the Young's modulus of the specimen fabricated by the PCHP continuously decreased. Since there are much more defects in the specimen fabricated by PCHP, the damping property is higher than that by PCHIP. It was thought that the joint structure fabricated by PCHIP is stronger than that by PCHP.

4. CONCLUSIONS

A quasi-isostatic pressing method by using the SPS system was used for the fabrication of the metallic cellular material. The hollow sphere particles were sintered to metallic cellular material. Microstructure observation showed that PCHIP provided a well hot-isostatic-pressing sintering for the soft particles. A metallic cellular material with fine and equiaxed structure was obtained. The Young's modulus of the specimen fabricated by PCHIP is larger than that by PCHP.

ACKNOWLEDGEMENTS

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