

## Fabrication of metallic closed cellular materials by sintering of metal coated heat resisting particles

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A metallic closed cellular material for intelligent and smart materials and structures has been fabricated by sintering of metal coated powder particles. Powder particles of heat resisting polymer coated with nickel-phosphorus alloy layers using electro-less plating were pressed into pellets (green pellets) and sintered in vacuum at high temperature using an electric furnace or a spark plasma sintering system. Then a metallic closed cellular material containing organic material was then constructed. The cross-sections of the specimens fabricated by different conditions were observed using the scanning electron microscope. The compressive and damping tests were carried out to measure the mechanical property of this material. The results of the compressive tests show that this metallic closed cellular material has low Young's modulus but breaks brittlely. In addition, the specimens sintered at different conditions have different mechanical properties. The results of the damping tests show that the internal friction of this material is same as that of pure aluminum.

Keywords: Metallic closed cellular material, energy absorbability, smart structure, electro-less plating, sintering, isostatic pressing, internal friction.

### 1. INTRODUCTION

Many current researches have been studied to develop the intelligent materials, smart materials and smart structures. Particularly, passive and active damping functions are becoming increasingly important in terms of vibration control of the structures and energy absorbing system has been required to protect persons from injury during impact of accident. Therefore, these materials, which have high-energy absorbability and damping function are required.

Recently, cellular materials are receiving renewed attention as structural and functional materials. Cellular materials have unique thermal, acoustic and energy absorbing properties that can be combined with their structural efficiency<sup>1</sup>. Therefore, many kinds of cellular materials have been tested as energy absorbing and damping materials. Particularly, the closed cellular materials are thought to have many favorable properties and applications. However, there is a lack of technique to produce such fine closed cellular materials except for the gas forming<sup>2-7</sup>, the sintering of hollow powder particles<sup>8,9</sup> and the two-dimensional honeycomb structures<sup>1</sup>. Therefore, authors have developed a fabrication process of metallic closed cellular material containing organic materials for the intelligent materials or smart structures<sup>10</sup>. However, further investigation indicated that the material inside the cell walls was carbonized<sup>11</sup>.

In this study, to fabricate a metallic closed cellular material containing organic materials, heat resistant polymer particles coated with metal layer was used. The cross-section of this material was observed. The compressive and damping properties of this closed cellular material containing organic materials are measured. The utility of this material is also discussed.

### 2. CONCEPTUAL PROCESS

The metallic closed cellular material fabricating process<sup>10</sup> is shown in Fig. 1. The process is as follows: 1) Powdered polymer particles are coated with a metal layer using electro-less plating. 2) The powder particles are pressed into pellets (green compacts) by cold isostatic pressing. 3) After sintering at high temperature in a vacuum, the closed cellular material is produced.

### 3. EXPERIMENTS

#### 3.1 Preparing the metallic closed cellular material

A heat resisting polymer, polyimide, particles of 8.5  $\mu\text{m}$  diameter (Ube Industries LTD.) was selected for this study. These polyimide particles were coated with 0.2  $\mu\text{m}$  thick nickel-phosphorus alloy layers using electro-less plating. These particles were pressed into pellets (green compacts) with about 8 mm diameters and 8 mm long by isostatic pressing at 200MPa and 90 °C. After this, these green compacts were sintered for 1 h at 800 °C and 850 °C in a vacuum. Then metallic closed cellular materials containing organic materials were then fabricated.

#### 3.2 Characterization

The microstructure of the powder particles, the green compacts before sintering, the cross-sections after sintering and fracture surface were observed using a scanning electron microscope (SEM). To observe the cross-section of this material, the specimen was cut and the cross-section surface was polished using emery paper (#600) and then 0.05  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  powders. The damping tests were carried out to estimate the internal friction of this material. The measurement was carried out using an about 1.0mm thickness plate-type specimen and a free resonance vibration-type

equipment(JE-RT, Nihon Techno-Plus Co., Ltd.). The internal friction was calculated by the half width method.

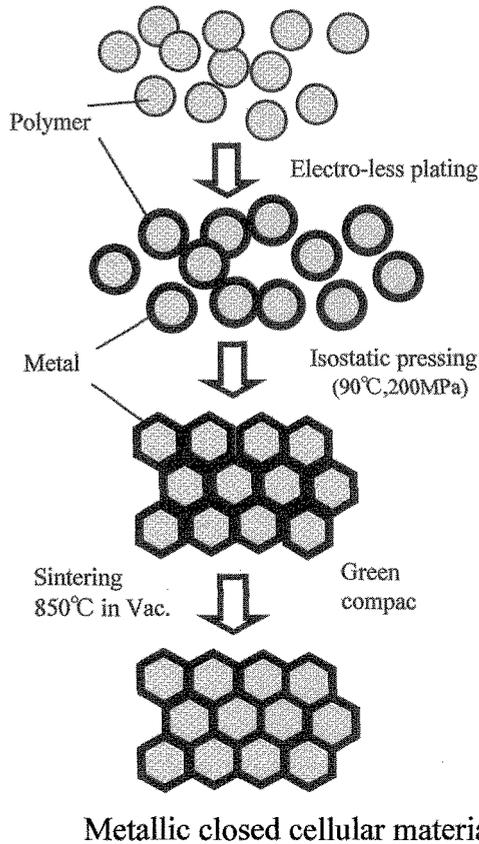


Fig. 1 Flow diagram of metallic closed cellular material fabricating process.

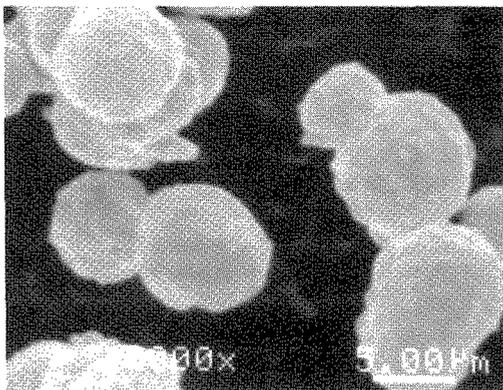


Fig.2 SEM image of polyimide particles coated by Ni-P alloy.

## 4. RESULTS

### 4.1 Micro-structural observation

Figure 2 shows an SEM image of the polyimide powder particles coated with Ni-P alloy layers and Fig. 3 shows an SEM image of the cross-section of the particle.

Figure 4 shows the green compact after cold isostatic pressing. The polyimide particles were deformed to polyhedrons and contacted each other. An SEM image of the cross-section of this material after sintering at 850°C is shown in Fig. 5. In this figure, the cell walls of the nickel-phosphorus alloy are observed as bright parts and the material inside the cell walls is observed as the darker parts.

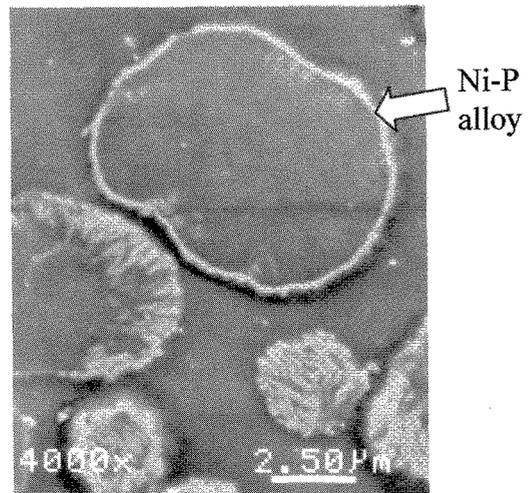


Fig.3 SEM image of cross-section of polyimide particles coated with Ni-P alloy layer.

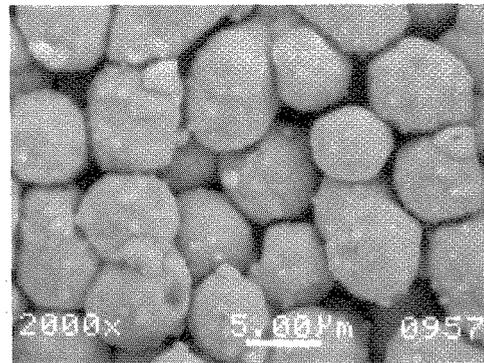


Fig.4. SEM image of green compact after isostatic pressing

### 4.2 Compressive test

Compressive tests were carried out at room temperature. A typical example of the compressive test results is shown in Fig. 6. Figure 6 shows stress-strain curves of the specimen sintered at 800°C and that sintered at 850°C. The stress-strain curve shows a linear elastic region, a long plateau where the stress gradually increases and a wavy region where the stress repeatedly decreases and increases. A specimen sintered at 800°C has higher strength than that of specimens sintered at 850°C.

### 4.3 Damping measurement

The internal friction of this material was measured by an elastometer (free resonance vibration-type). Figure 7 shows one example of the result of the internal friction measurement of the specimen sintered at 850°C. The internal friction was calculated by half-value method. The internal friction of this material is from  $5.77 \times 10^{-3}$  to  $1.56 \times 10^{-2}$ . To compare with these results, the internal friction of pure aluminum was measured and its value was about  $5.25 \times 10^{-3}$ . These results suggest that the internal friction is almost same as that of pure aluminum at room temperature.

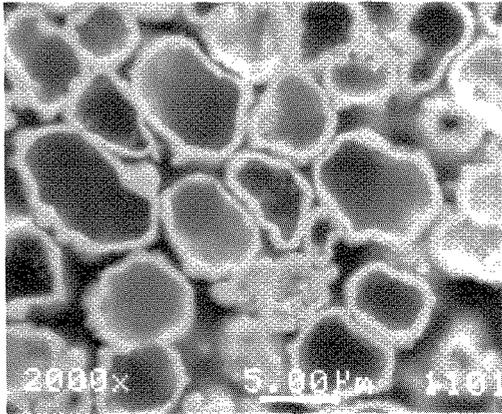


Fig. 5. Cross-section of the metallic closed cellular material.

## 5. DISCUSSION

### 5.1 Metallic closed cellular material

A metallic closed cellular material has been fabricated in this study. The density of this material was about  $3.1 \text{ g/cm}^3$  and little larger than that of pure aluminum. As Fig. 5 shows that cell walls of a nickel-phosphorus alloy are observed as bright parts and the material inside the cell walls is observed as darker parts. This result indicates that the organic material remains inside of the cell walls after heat treatment and this metallic closed cellular material containing the organic material can be produced using this technique.

### 5.2 Energy absorption

As shown in Fig. 6, the stress-strain curve has a linear elastic region and a wavy region. It is thought that the presence of the plateau in the compressive stress-strain curve is responsible for the high-energy absorption. However, any plateau region could not be observed and a long wavy region in the stress-strain curves was observed.

As Fig. 5 shows, some defects and spaces are shown between neighboring particles. During the linear elastic region, a few cracks occur in the direction parallel to the stress axis. It is postulated that the fracture initiates from a defect in this material. Therefore, if this metallic closed cellular material has only few defects, the plateau area of the stress-strain curve will occur and continue longer during the compressive test and this material has large energy absorption.

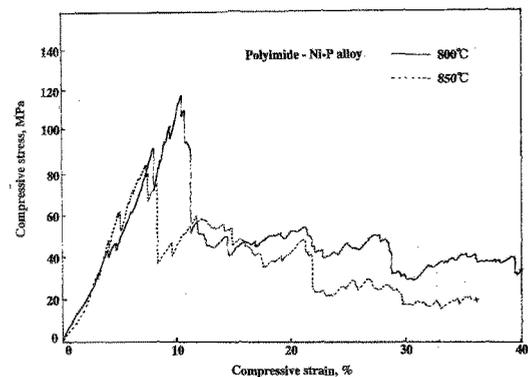


Fig.6 Stress-strain curves of metallic closed cellular materials specimen during compressive tests.

### 5.3 Young's Modulus

Young's modulus of this material was measured using the data in linear elastic region for each specimen, and the relationship between Young's modulus of this material is 1630MPa (sintered at 800°C) and 1818MPa (sintered at 850°C), respectively. Young's modulus of the specimen sintered at higher temperature is larger than that of the specimen sintered at lower temperature. Compare with the result of another cellular materials<sup>11</sup>, Young's modulus is almost same but the stress-strain curves show that this material looks brittle.

### 5.4 Internal friction

The internal friction of this material is almost same as that of pure aluminum and larger than that of the metallic closed cellular materials containing carbonized polystyrene.<sup>11)</sup> Figure 8 shows the relationship between the temperature and internal frictions. The internal friction increase due to increasing of the temperature and it is largest around the temperature from 40 to 90°C. This result shows the internal friction (damping property) changes according to the changing of the temperature.

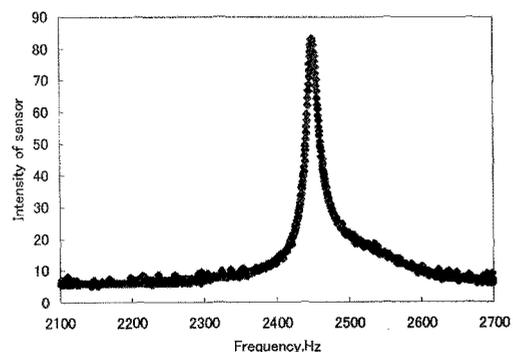


Fig.7. Relationship between the frequency and the intensity of the sensor.

This phenomenon occurs by the interaction between the materials inside the cells and that of cell walls. These results suggest that this material can be utilized as a passive damping material.

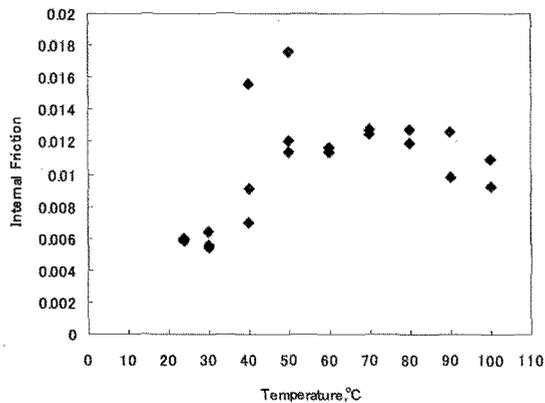


Fig.8. Relationship between the temperature and the internal friction.

#### 6. CONCLUSION

A metallic closed cellular material containing organic materials has been developed. This metallic cellular material was light and has the large internal friction and capability of large energy absorption. The obtained results emphasized that this metallic closed cellular material can be utilized as the energy absorbing material and passive damping material.

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(Received October 10, 2003; Accepted March 20, 2004)