Functionally Graded Al Foam Fabricated by Sintering and Dissolution Process with Remaining Spacers

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Functionally graded materials composed of Al foam and Al/spacer composites were fabricated by a sintering dissolution process, in which removal of the spacers was stopped during the dissolution process. The deformation of the material started in the low-strength Al foam layer then in the high-strength layer with the remaining spacers. [doi:10.2320/matertrans.M2015404]

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1. Introduction

Open-cell metallic foam is expected to be used for components in automobiles owing to its superior shock absorbance and light weight¹⁾. A space holder method is a promising process for fabricating open-cell metallic foam owing to its control of the pore structures. In this process, a metal/spacer composite is first fabricated by a casting process²⁾ or by a powder metallurgy process^{3–5)}. Thereafter, the spacers are removed from the composite, resulting in the formation of pores in the metal. During the removal process of the spacers, two regions exist. One is the metallic foam region in which spacers have already been removed, and the other is the region with remaining spacers (hereinafter “spacer region”). In the spacer region, it is considered that the metallic foam is reinforced by the spacer particles. Previous studies⁶⁾ indicated that the compression deformation of a metal/spacer composite, which has both a metallic foam region and a spacer region, first occurs in the metallic foam layer and then in the spacer layer. This deformation behavior is similar to that of functionally graded (FG) metallic foam, in which the compression properties vary with the position and deformation sequentially occurs from the lower-strength layers to the higher-strength layers^{7,8)}. Also, it was shown that the compression properties of a metal/spacer composite change with the percentage of the spacer region in the composite. Therefore, it is expected that FG metallic foam with a gradual change in its mechanical properties, which depend on the removal time of the spacers, can be realized by forming a metal/spacer composite.

In this study, an FG aluminum (Al)/sodium chloride (NaCl) composite was fabricated by a sintering and dissolution process (SDP)³⁾, using NaCl as the spacers, so that the removal of spacers is easily achieved by simply immersing the Al/NaCl composite in water. First, the Al/NaCl composite was observed nondestructively by X-ray computed tomography (CT) during the spacer removal process to reveal the changes in the three-dimensional region in which NaCl remained (hereinafter “NaCl region”) to confirm that two layers, i.e., an Al foam layer and a NaCl layer, actually exist in

the FG material. Second, a compression test specimen with a remaining NaCl weight ratio of approximately 10% was fabricated. The deformation behavior during compression tests of the specimen was nondestructively observed by X-ray CT and the mechanical properties of the FG Al/NaCl composite were investigated. From these nondestructive observations, the effect of the NaCl region in the Al/NaCl composite on the mechanical properties and deformation behavior of the Al foam was investigated.

2. Experimental Procedure

Commercially available as-received pure Al (99.9% purity, average diameter of 20 μm) and NaCl (99.9% purity, diameter range from 300 μm to 425 μm) powders were thoroughly mixed with a NaCl volume fraction of 74%. The mixture was then subjected to spark plasma sintering using an SPS-515L (Sumitomo Coal Mining Co., Ltd.). The sintering temperature, pressure, and time were fixed to 843 K, 20 MPa, and 10 min, respectively⁹⁾. Two cylindrical Al/NaCl composite specimens with dimensions of $\phi 20 \text{ mm} \times 13.8 \text{ mm}$ were obtained.

NaCl was removed from the specimen by leaching in a water bath. The water temperature was set at 333 K. The amount of remaining NaCl φ was determined as the weight fraction of the NaCl remaining in the specimen relative to the total amount of NaCl in the initial specimen as follows;

$$\varphi = \left(1 - \frac{W_{\text{init}} - W_{\text{comp}}}{W_{\text{init}} \times f} \right) \times 100,$$

where W_{init} is initial weight of the as-sintered specimen before the removal process, W_{comp} is the weight of the specimen during the removal process, and f is the weight of NaCl as a fraction of the initial weight of the as-sintered specimen, i.e., $W_{\text{init}} \times f$ is the weight of NaCl in the initial as-sintered specimen.

One of the specimens was subjected to a stepwise removal process. At fixed intervals during the removal process, the specimen was pulled out of the water bath and dried, then an X-ray CT observation of the remaining NaCl was conducted and the specimen was weighted, then the next removal process was conducted. The relationship between the leaching

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Fig. 1 X-ray CT image of the specimen ($\varphi = 11.1\%$).

time and the weight fraction of the remaining NaCl was obtained along with the three-dimensional NaCl distribution in the specimen during the removal process.

The other specimen was subjected to an uninterrupted removal process to obtain a compression test specimen with $\varphi = 11.1\%$. This specimen was subjected to a stepwise compression test. The deformation behaviors of the specimen and the remaining NaCl during the compression tests were nondestructively observed by X-ray CT.

X-ray CT observations were conducted using an SMX-225CT microfocus X-ray CT system (Shimadzu Corporation)^{7,8)}. The resolution of each CT image was 512×512 and the pixel length was approximately $70 \mu\text{m}$. Figure 1 shows a representative two-dimensional cross-sectional X-ray CT image of the specimen. Gray regions indicate the cell walls of Al foam and NaCl, and black regions indicate pores. To obtain three-dimensional images of the NaCl region in the specimens, a voxel model of each specimen was constructed using VOXELCON image-processing software (Quint Corporation). First, two-dimensional cross-sectional X-ray CT images were stacked, then an appropriate threshold was set to distinguish NaCl/Al and pore voxels to obtain the voxel model.

The specimen ($\varphi = 11.1\%$) was loaded in a stepwise manner during the compression tests. After each step of compression, the specimen was unloaded, an X-ray CT observation was conducted, then the next compression was conducted. The compression tests were carried out using an Autograph AG-100kNG universal testing machine (Shimadzu Corporation) at a strain rate of $3.3 \times 10^{-3} \text{ s}^{-1}$. After each loading and unloading process, the specimen was placed in the X-ray CT equipment and nondestructive observations were conducted. Series of deformation images were observed with an interval of 2.5% compression strain for a total compression strain of 0–40% and with an interval of 10% compression strain for a total compression strain of 40–80%. After X-ray CT observation, the specimen was returned to the universal testing machine and subjected to the next compression.

3. Experimental Results and Discussion

Figure 2(a) shows the relationship between the leaching time t and φ . The specimen before the removal process (i.e., $\varphi = 100\%$) is also shown in the figure. Figures 2(b)–(g) show three-dimensional images of the NaCl region during the removal process. The remaining NaCl appeared as a white region, and the cell walls of the pores appeared as a faint white region. The size of the NaCl region gradually decreased with

Fig. 2 (a) Relationship between ratio of remaining NaCl and leaching time along with appearance of as-sintered Al/NaCl composite before removal process. (b)–(g) NaCl region in the specimen with (b) $\varphi = 59.2\%$, (c) $\varphi = 35.2\%$, (d) $\varphi = 16.2\%$, (e) $\varphi = 10.2\%$, (f) $\varphi = 1.5\%$, and (g) $\varphi = 0\%$.

increasing leaching time. The reduction of the size of the NaCl region started from the outside of the specimen, where water could directly come into contact with NaCl, whereas NaCl remained at the center part of the specimen. Although the area of NaCl in the NaCl layer gradually decreased and the area of Al foam increased with increasing leaching time, two layers, Al foam and NaCl layers, can be clearly observed.

Figure 3 shows sequential deformation images of the specimen ($\varphi = 11.1\%$) during the compression tests. The deformation first occurred in the upper and lower Al foam layers and then in the NaCl layer. Figure 4 shows the stress-strain curve of the specimen ($\varphi = 11.1\%$) and the Young's modulus, which was evaluated from the slope of the stress-strain curve obtained in the unloaded state during the stepwise compression tests, along with the stress-strain curve of the Al foam ($\varphi = 0\%$)⁶⁾. Figure 5 shows the normalized volume of the remaining NaCl at each compression strain. The stress-strain curve of the specimen was similar to that of the Al foam up to $\varepsilon = 20\%$. In this stage, only the upper and lower Al foam layers deformed (Fig. 3) and the volume of NaCl hardly decreased (Fig. 5). Therefore, almost the same plateau region as that for Al foam can be observed, as well as an almost constant Young's modulus. At $\varepsilon = 20$ –40%, the stress increased gradually apart from the plateau regions of the Al foam. However, the upper and lower Al foam layers still deformed, and the

Fig. 3 Deformation images during compression tests of Al/NaCl composite ($\varphi = 11.1\%$).

Fig. 4 Stress-strain curve for Al/NaCl composite ($\varphi = 11.1\%$) and Young's modulus plotted against strain along with stress-strain curve of Al foam ($\varphi = 0\%$)⁶⁾.

Fig. 5 Volume of NaCl region at each compression strain.

NaCl region approximately retained its initial shape and volume (Figs. 3 and 5), i.e., the NaCl layer hardly deformed. Therefore, it is considered that densification of the upper and lower Al foam layers occurred, and thus, the stress and Young's modulus increased. At $\varepsilon = 40$ – 60% , the stress increased markedly. The NaCl layer started to deform (Fig. 3) and the volume of NaCl rapidly decreased (Fig. 5). The Young's modulus exhibited an almost constant value that was higher than the value at $\varepsilon < 20\%$. This tendency was consistent with the results of other FG Al foam fabricated by the

precursor foaming process⁷⁾. Consequently, it was found that percentage of NaCl remaining in the Al/NaCl composite can be used to control its deformation behavior and mechanical properties, and thus produce tailored FG materials. Clearly, further studies are necessary, such as to compare Young's modulus between the FG Al foam and Al foam ($\varphi = 0\%$), to reveal the accurate mechanical properties of the FG Al foam.

4. Conclusion

In this study, an FG material composed of an Al foam region and an Al/NaCl composite region was fabricated by a sintering and dissolution process. From the X-ray CT observation carried out during the removal of NaCl spacers from the Al/NaCl composite, it was shown that the size of the NaCl region gradually decreased with increasing leaching time. Two layers, Al foam and NaCl layers, were clearly observed in the composite. From the compression test of the Al/NaCl composite ($\varphi = 11.1\%$), it was found that the deformation first occurred in the Al foam layer with low strength and then in the NaCl layer with high strength. Therefore, the percentage of NaCl remaining in the Al/NaCl composite can be used to control its deformation behavior and mechanical properties, and thus produce tailored FG materials.

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