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What is This?
Manufacturing process of dispersed intermetallic compounds Al alloy composites by using porous nickel

YB Choi, K Matsugi and G Sasaki

Abstract
A new process is proposed to fabricate an intermetallic compound-reinforced aluminum alloy matrix composite using the reaction between porous nickel and molten aluminum alloy. The intermetallic compound-reinforced aluminum alloy composite was manufactured with the infiltration process method. Porous nickel reacted with the molten aluminum alloy at 973 K, and the intermetallic compound of Al$_3$Ni was generated on the surface of the porous nickel. The generated intermetallic compound Al$_3$Ni is delaminated due to different thermal expansion coefficient with nickel and moves in the direction of aluminum matrix. The effects of processing variables such as specific surface area of porous nickel, holding time after infiltrated molten aluminum alloy, and cooling rate on the formation and dispersion behavior of Al$_3$Ni were investigated.

Keywords
Porous, nickel, intermetallic, compound, Al$_3$Ni, manufacturing, process

Introduction
Recently, the conversion to aluminum alloy materials from cast iron materials has been increasing. And, advanced features of the aluminum alloy are demanded. The composites that are strengthened with ceramic particles in aluminum alloy are developed. It is practically applied for brake disk and piston in mobile parts in the industrial field. Generally, manufacture methods of a ceramic particles dispersed composite include Powder metallurgy process, Melting stirring method, and Casting method.

Powder metallurgy process requires high cost. Composites fabricated by powder metallurgy process have low density. Melting agitating method is the process of using the reactivity of melting aluminum and an additive element to manufacture a reaction product. However, fabrication of a complicated-shaped composite is difficult, and the control of the dispersibility of an intermetallic compound is difficult for this method. Casting method is a simple fabrication process to make composite. When wettability of an additive element and aluminum is not good, the dispersibility of particles in matrix is a problem. Moreover, when a composite material is applied as piston head or ring portion by the conventional producing method, it raises more problems.

Whereas, in this research, ceramic particle-reinforced composite is applied to the piston head and ring portion of automobile engine parts, which is why a development of a new process is proposed. To manufacture intermetallic compound-reinforced composite, melted aluminum alloy is infiltrated into porous nickel by low-pressure infiltration method. Intermetallic compound is generated by the reaction between aluminum and nickel. This research investigated the reaction behavior of the intermetallic compound between aluminum and porous nickel at two
different specific surface areas. Furthermore, the influence of cooling rate and holding time after molten Al alloy was infiltrated were investigated in the microstructure of intermetallic compound-reinforced composite.

Materials and experiment processes

A336 alloy (Al-12mass%Si-1mass%Ni-1 mass%Cu-1mass%Mg) was used as matrix in this experiment. Preform was porous nickel (Toyama Sumitomo Electric Co., Ltd.). Volume ratio of porous nickel is 4–6%. Figure 1 shows the scanning electron microscope (SEM) images of porous nickel. Porous nickel has three-dimensional network structure like sponge, large surface area, and it is easy to be machined. Porous nickel with two kinds of specific surface areas ((a) 1250 mm²/mm³ and (b) 2800 mm²/mm³) were used in the experiment to examine the reactive behavior of the intermetallic compound. The properties of porous nickel were shown in Table 1. Low-pressure infiltration method was used to fabricate the composites. Fabricating conditions were altered by changing the holding time after infiltration of molten Al alloy and cooling rate. Holding time was changed from 1s to 1200 s at 0.1 MPa pressure. Cooling rate was changed from 0.08 to 1.32 K/min after holding for 10 min. Conditions of cooling rate are shown in Figure 2. Cooling rate is controlled by the solid-phase temperature of aluminum alloy.

Microstructure of the composites was observed by SEM. Aspect ratio (ratio of length and width) and area rate of the intermetallic compounds by specific surface area of porous nickel were measured. Energy-dispersive X-ray spectroscopy (EDX) was used to determine the phase compositions. Micro-Vickers hardness was evaluated on the intermetallic compounds and matrix as well.

Results and discussion

Reaction of porous nickel and molten Al alloy

Figure 3 shows the microstructure SEM images of porous nickel (specific surface area: 1250 mm²/mm³) reinforced A366 alloy composite. Molten Al alloy was infiltrated to porous nickel by using low-pressure infiltration at 0.1 MPa.

EDX analysis confirmed the existence of Al₃Ni phase, nickel phase at the central section of porous nickel, and the presence of Al₃Ni₂ phase between the Al₃Ni phase and nickel phase. Other intermetallic compounds were not detected. The phase-formation sequence was described in the following equation:

$$3\text{Al} + \text{Ni} \rightarrow \text{Al}_3\text{Ni} - 190 \text{kJ/mol}$$

These observations were consistent with the report by Hibino on the rate of formation of Ni-Al intermetallic compounds. As seen in Figure 3, the porous nickel does not instantly react and change into Al₃Ni. On the contrary, the reaction between the molten Al alloy and porous nickel gradually proceeds, working toward the center of the porous nickel from the outer surface, and fine Al₃Ni are dispersed into the matrix. In light of the densities of the Ni, Al₃Ni₂, and Al₃Ni, i.e. 8.9, 4.8,

<table>
<thead>
<tr>
<th>Samples</th>
<th>Cell size (mm)</th>
<th>Volume fraction (vol%)</th>
<th>Thickness (mm)</th>
<th>Specific Surface area (mm²/mm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous nickel</td>
<td>0.73–0.98</td>
<td>4–6</td>
<td>10</td>
<td>1250</td>
</tr>
<tr>
<td>Porous nickel</td>
<td>0.45–0.58</td>
<td>4–6</td>
<td>5</td>
<td>2800</td>
</tr>
</tbody>
</table>

Figure 1. SEM images of specific surface area of porous nickel (a) 1250 mm²/mm³ and (b) 2800 mm²/mm³.
and 4.0 kg/m$^3$, the porous nickel is thought to undergo an expansion reaction with the molten Al alloy.$^9$

**Effect of specific surface area of porous nickel**

Figure 4 shows the microstructure SEM images of Al$_3$Ni/A366 composites with specific surface area: (a) 1250 and (b) 2800 mm$^2$/mm$^3$ under fabrication conditions of molten Al alloy of 973 K and 0.1 MPa. As seen in Figure 4(a), most of the fine Al$_3$Ni intermetallic compounds are homogeneously dispersed into the matrix. But shape of the needle-like Al$_3$Ni was observed too. As for the result of the area rate of the intermetallic compound and unreacted nickel in the measured area of 104 mm$^2$, 14.92% of the intermetallic compound and 2.70% of the unreacted nickel were observed. And, numerous pores were observed in the composites.

As seen in Figure 4(b), most of the fine Al$_3$Ni intermetallic compounds are dispersed into the matrix compared with fabricated composite using porous nickel with low-specific surface area. In the case of intermetallic compound using high-specific surface area, the result of the area rate of the intermetallic compound and unreacted nickel in the measured area of 104 mm$^2$, 28.09% of the intermetallic compound and 2.28% of unreacted nickel were observed.

In light of the specific surface area of the porous nickel, i.e. 1250 and 2800 mm$^2$/mm$^3$, the intermetallic compound produced by the porous nickel of a high-specific surface area (2800 mm$^2$/mm$^3$) is increased.
compared with the porous nickel of a low-specific surface (1250 mm\(^2\)/mm\(^3\)). It was also observed that the quantity of unreacted nickel decreased.

Figure 5 shows the results of measured shape of Al\(_3\)Ni in the composite with specific surface area of porous: 1250 (a) and 2800 (b) under fabrication conditions of 973 K and 0.1 MPa. Aspect ratio (ratio of length/width) of Al\(_3\)Ni intermetallic compound was measured.

There are two kinds of Al\(_3\)Ni/A366 composite having the aspect ratio, three or less of Al\(_3\)Ni (granular shape) having a lot. However, aspect ratio, three or more (shape of needle-like) was observed having a lot for Al\(_3\)Ni/A366 composite with the porous nickel (specific surface area: 1250 mm\(^2\)/mm\(^3\)).

**Effect of holding time after infiltrated molten Al alloy**

Figure 6 shows the microstructure of Al\(_3\)Ni/A366 composites (specific surface area: 1250 and 2800 mm\(^2\)/mm\(^3\)) fabricated at various holding time after infiltrated molten Al alloy. Applied pressure and temperature of molten alloy were 0.1 MPa and 973 K with furnace cooling.

As a result of observing the microstructure of the fabricated composite using two kinds of porous nickel with holding time of 1 s, there were few intermetallic compounds and mostly were unreacted nickel.

When holding time after infiltrated molten Al alloy is controlled at 600 s, all of the porous nickel are reacted with molten Al alloy to form fine Al\(_3\)Ni intermetallic compounds that dispersed into the matrix, and the morphology of Al\(_3\)Ni was granular (Figure 6(b) and (e)). In addition, unreacted nickel was not observed in the composite. However, the intermetallic compound using a high-specific surface area (2800 mm\(^2\)/mm\(^3\)) is more dispersed than that of the porous nickel of a low-specific surface (1250 mm\(^2\)/mm\(^3\)). In the case of holding time of 1200 s, needle-like-shaped coarse Al\(_3\)Ni was observed. Figure 6(c) and (f) also shows the increase of the size of the Al\(_3\)Ni in parallel with the increasing of the holding time between nickel and molten Al alloy. In order to control fine intermetallic compound and distribution of the intermetallic compound, the result is that it is better to use the high-specific surface area and holding time for 600 s.

**Effect of cooling rate**

Figure 7 shows the microstructure of Al\(_3\)Ni/A366 composites with cooling rate: (a) furnace cooling, (b) air cooling, and (c) forced air cooling under fabrication conditions of 973 K, reaction time of 600 s, and...
0.1 MPa. As seen in Figure 6(a), all of the porous nickel reacted with Al alloy to form Al$_3$Ni intermetallic compounds and Al$_3$Ni that dispersed into the matrix, and the morphology of Al$_3$Ni was granular (Figure 6(a)). Most of the fine Al$_3$Ni intermetallic compounds were homogeneously dispersed inside the matrix.

However, in the case of air cooling and a forced air cooling, a little fine intermetallic compounds were observed inside matrix (Figure 6(b) and (c)), and most of the nickel has not reacted with aluminum. The form of the porous nickel remains inside of matrix.

Although intermetallic compound by the reaction between nickel and molten Al alloy is generated inside the matrix with 600’s of holding time, the amount of intermetallic compounds is few. Set the cooling rate as the furnace cooling will contribute to the formation of the intermetallic compounds greatly.

**Micro-Vickers hardness**

Figure 8 shows the results the micro-Vickers hardness of the Al$_3$Ni intermetallic compound and a matrix.
The fabrication conditions of composites are applied pressure of 0.1 MPa, the 973 K temperature of molten Al alloy, holding time of 600 s, and furnace cooling using the porous nickel of specific surface area 2800 mm²/mm³.

Figure 8(a) shows the SEM image of the impression of Al₃Ni and (b) shows the SEM image of the impression of the matrix. The micro-Vickers hardness of Al₃Ni intermetallic compound and matrix are 675 and 74 Hv, respectively. Since the Al₃Ni/A336 composite (specific surface area: 2800 mm²/mm³) contains 28.9% of intermetallic compound, which is expected to possess the best potential to improve the mechanical properties of the materials.

Conclusions

New process is proposed to fabricate intermetallic compound-reinforced Al alloy matrix composites by the reaction between porous nickel and molten Al alloy. The fabrication process to obtain fine intermetallic compound from a porous nickel reacts to aluminum was developed. The important results are listed below.

1. The intermetallic compounds formed by the reaction of porous nickel with Al alloy are produced by low-pressure infiltration. Fine and granular Al₃Ni has been distributed in the Al alloy matrix under holding time of 600 s after infiltrated molten Al alloy. However, even though intermetallic compound is generated inside the matrix at holding time of 600 s, the amount of generated intermetallic compounds is few.
2. Al₃Ni/Al alloy composite fabricated by furnace cooling was homogeneously dispersed inside the matrix finely. However, in the case of air cooling and forced air cooling, a few fine intermetallic compounds were observed inside the matrix. Cooling conditions were important to control the amount of intermetallic compounds. The amount of intermetallic compound increased with the increasing cooling rate.
3. The aspect ratio of the Al₃Ni was observed, three or less of Al₃Ni. Most of Al₃Ni has the aspect ratio (1–3) of particulate shape. The reaction of porous nickel and Al alloy was fabricate particle-shaped intermetallic compound-reinforced composite.
4. Micro-Vickers hardness of Al₃Ni intermetallic compound and matrix were 675 and 74 Hv, respectively. Al₃Ni/A336 composite (specific surface: 2800 mm²/mm³) contained area ratio and 28.9% of intermetallic compound, and the Al₃Ni/A366 composite is expected to improve the mechanical properties with the most potential.

References


