Manufacturing process of particle intermetallic compound reinforced Al base composites by infiltration-reaction method

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Abstract
A new process is proposed to fabricate an aluminum alloy matrix composite dispersed with intermetallic compound particles by using the reaction between porous nickel and molten aluminum alloy. The intermetallic compound particles reinforced aluminum alloy matrix composite was manufactured with the infiltration-reaction method. Four different specific surface areas of porous nickel were used to fabricate the composites and then the porosity inside composites were investigated. Porous nickel reacted with molten aluminum alloy at 973 K, and the intermetallic compound, Al3Ni was generated on the surface of the porous nickel. The generated intermetallic compound Al3Ni, was delaminated according to the difference of thermal expanion coefficient with nickel. And the intermetallic compounds moves in the direction of aluminum matrix. The area fraction of the intermetallic compounds increased with the increasing specific surface area of porous nickel. In addition, the hardness of composite increased by the increasing specific surface area of porous nickel.

Key words : Metal-matrix composites, Intermetallic compound, Pressure infiltration, Reaction, Mechanical properties

1. 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 which are strengthened with ceramic particles in aluminum alloy are developed. It is applied in practical for brake disk and piston in mobile parts in the industrial field (Choi Y. B., et al., 2006, Lee M. H., et al., 2014). Generally, manufacture methods of a ceramic particles dispersed composite include powder metallurgy process (Izadi H., et al., 2013), stir casting (Bharath V., et al., 2014) and squeeze casting method (Vijayaram T. R., et al., 2006), etc.

Powder metallurgy process requires high cost. 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 shape composite is difficult. Casting method is 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 many problems. Whereas, in this research ceramic particles reinforced composite is applied to the piston head and ring portion of piston parts, which is why a development of a new process is purposed. To manufacture intermetallic compound reinforced composite, molten aluminum alloy is infiltrated into porous nickel by applying low-pressure infiltration - reaction method. Intermetallic compound is generated by the reaction between aluminum and nickel.

The objective of this research is to investigate the effects of the specific surface area on the intermetallic compounds formed in the composites by infiltration-reaction methods. Therefore, the research investigated on the reaction of porous nickel and molten Al alloy, the amounts of the intermetallic compounds by different specific surface area of porous nickel and the aspect ratio of intermetallic compounds under 1250m²/m³, 2800m²/m³, 5800m²/m³ and > 5800m²/m³, respectively. In addition, the porosity inside composites and hardness were also investigated.
2. Materials and Experimental procedure

A366 alloy in ASTM, which composition of Al-12Si-1Ni-1Cu-1Mg (mass %), was used as matrix in this experiment. Preform was porous nickel (Toyama Sumitomo Electric Co., Ltd.). Volume fraction of porous nickel is 4~6%. Figure 1 shows SEM images of porous nickel. Porous nickel has three-dimensional network structure like sponge, large surface area and it is easy to machine. Porous nickels with four different kinds of specific surface areas (a: 1250 m$^2$/m$^3$, b: 2800 m$^2$/m$^3$, c: 5800 m$^2$/m$^3$ and d: > 5800 m$^2$/m$^3$) 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 and reaction method was used to fabricate the composites. Temperature of molten Al alloy and applied pressure to molten Al alloy were 973K and 0.1MPa. This fabrication condition is the one proved by previous studies which were most suitable for generation of the intermetallic compound (Choi Y. B., et al., 2013). Holding time, after applied 0.1MPa pressure, is 10 minutes. Microstructure of the composites was observed by Optical Microscope (OM) and Scanning Electron Microscope (SEM). And the area fraction, counts, aspect ratio (ratio of length and width) and porosity inside matrix were measured by picture analysis using the image pro-plus. Area analysis of each composite is conducted on the basis of area 480mm$^2$.

Energy dispersive X-ray spectroscopy (EDX), was used to determine the phase compositions. Hardness was investigated by Micro-Vickers hardness and Rockwell hardness (A scale) with 120° diamond spheroconical indenter.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Cell size (mm)</th>
<th>Volume fraction, % (vol.)</th>
<th>Specific Surface area (m$^2$/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous nickel</td>
<td>0.98</td>
<td>4-6</td>
<td>1250</td>
</tr>
<tr>
<td></td>
<td>0.55</td>
<td>4-6</td>
<td>2800</td>
</tr>
<tr>
<td></td>
<td>0.45</td>
<td>4-6</td>
<td>5800</td>
</tr>
<tr>
<td></td>
<td>0.35</td>
<td>4-6</td>
<td>&gt;5800</td>
</tr>
</tbody>
</table>

![Fig. 1 SEM images of porous nickel (Specific surface area: (a) 1250 m$^2$/m$^3$, (b) 2800 m$^2$/m$^3$, (c) 5800 m$^2$/m$^3$ and (d) >5800 m$^2$/m$^3$)](image)

3. Results and discussions

3.1 Reaction of porous nickel and molten Al alloy

Figure 2 shows SEM images of porous nickel (specific surface area: 1250 m$^2$/m$^3$) inside matrix. Molten Al alloy of 973K was infiltrated to porous nickel by using low pressure infiltration casting at 0.1MPa and then was reacted between nickel and aluminum. EDX analysis confirmed the existence of Al$_3$Ni phase (darkest gray areas in Figure 2), nickel phase at the central section of porous nickel (brightest color areas in Figure 2) and an Al$_3$Ni$_2$ phase between the Al$_3$Ni and nickel. Other intermetallic compounds were not detected. The phase formation sequence was described as following equation: (Kubaschewski O., et al., 1993)

$$3\text{Al} + \text{Ni} \rightarrow \text{Al}_3\text{Ni} - 190\text{kJ/mol} \quad (1)$$

These observations were consistent with the report by Hibino on the rate of formation of Ni-Al intermetallic compounds (Hibino A., 1993)

As seen in Figure 2, the porous nickel does not instantly react and change to Al$_3$Ni. On the contrary, the reaction
between the molten Al alloy and porous nickel gradually proceeds, working towards the center of the porous nickel from the outer surface and fine Al$_3$Ni are dispersed into the matrix. In lightening of the densities of the Ni, Al$_3$Ni$_2$ and Al$_3$Ni, i.e., 8.9, 4.8 and 4.0 kg/m$^3$, the porous nickel is thought to undergo an expansion reaction with the molten Al alloy.

### 3.2 Effect of the specific surface area of porous nickel

Figure 3 shows microstructure of Al matrix composites dispersed intermetallic compound with specific surface area of porous: a) 1250 m$^2$/m$^3$, b) 2800 m$^2$/m$^3$, c) 5800 m$^2$/m$^3$ and d) >5800 m$^2$/m$^3$. As a result of observing the microstructure of the composites manufactured using four kinds of porous nickel, on the whole, the intermetallic compound were distributed inside matrix by the reaction of a porous nickel and aluminum. However, the microstructure observed from the composites fabricated with low specific surface area (in figure 3(a) 1250 m$^2$/m$^3$) that almost all porous nickel were changed to intermetallic compound. The area fraction of the generated intermetallic compound is about 15%. In addition, the portion which remains as porous nickel body without delamination from the porous surface was also observed. However, the area fraction of the un-reacted nickel was 2.8%. And most of the fine Al$_3$Ni intermetallic compounds (below 0.1mm$^2$ calculated area) were homogeneously dispersed inside matrix. But shape of the needle-like Al$_3$Ni was observed too. And numerous pores were observed in the composites. The microstructure when specific surface area is high (in figure 3(d) >5800 m$^2$/m$^3$), more fine intermetallic compounds were distributed as compared with other materials in figure 3(a), (b) and (c). The fine intermetallic compounds increased with increasing specific surface area of porous nickel. In addition, based on the figure 3(d) and figure 4, it showed that the area fraction (%) of intermetallic compound Al$_3$Ni was about 28.2%, which is almost twice of figure 3(a). Furthermore, the area fraction of un-reacted nickel reached a minimum value of 0 % when the specific surface was >5800 m$^2$/m$^3$. Results valus of area fractions of Al$_3$Ni and Un-reacted nickel was showing in table 2. The results indicate that higher the specific surface area of porous nickel is, larger the contact surface with molten Al is, and it provided further reaction of nickel with Al.

**Table 2 Area fraction of intermetallic compound and un-reacted nickel in composite.**

<table>
<thead>
<tr>
<th>Specific Surface area (m$^2$/m$^3$)</th>
<th>1250</th>
<th>2800</th>
<th>5800</th>
<th>&gt;5800</th>
</tr>
</thead>
<tbody>
<tr>
<td>Areafraction, %</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Intermetallic compound, Al$_3$Ni</td>
<td>15.01±2.71</td>
<td>20.18±1.99</td>
<td>26.68±4.29</td>
<td>28.22±2.13</td>
</tr>
<tr>
<td>Un-reacted nickel</td>
<td>2.80±0.08</td>
<td>1.50±0.78</td>
<td>0.21±0.08</td>
<td>0</td>
</tr>
</tbody>
</table>

Fig. 3 Microstructure of Al matrix composites dispersed intermetallic compound fabricated at 973K and 0.1MPa under each specific surface area of porous: (a) 1250 m$^2$/m$^3$, (b) 2800 m$^2$/m$^3$, (c) 5800 m$^2$/m$^3$ and (d) >5800 m$^2$/m$^3$
3.3 Aspect ratio and size of Al₃Ni, intermetallic compounds

Figure 5(a) shows results of measuring shape of Al₃Ni in composite with different specific surface area of porous: a) 1250 m²/m³, b) 2800 m²/m³, c) 5800 m²/m³ and d) >5800 m²/m³ under fabrication conditions of 973K and 0.1MPa. Aspect ratio (length/width ratio) of Al₃Ni, intermetallic compounds was measured. The result of aspect ratio when specific surface area is higher (>5800 m²/m³), intermetallic compound of 95.7% from an overall rate was observed for aspect ratio of 1 (granular-shaped, over 0.1mm² calculated area). In about 62.4% or more, the shapes of the granular-shaped intermetallic compounds exist compared with the specific surface area 1250 m²/m³. When specific surface area is low, Needle-like shaped intermetallic compounds with aspect ratio of 3 or more exist mostly. Size of Al₃Ni, intermetallic compound was reflected in figure 5(b). The size of Al₃Ni was showing a decreasing trend with the increasing of specific surface area, and reached to its maximum at >5800 m²/m³ with the counts of about 30µm. The results indicate that higher the specific surface area of porous nickel is, larger the contact surface of porous nickel with molten Al is, and it provided further reaction of Ni with Al. This result is that 95.7% of a granular-shaped intermetallic compound exists if the shown specific surface area increases, and that number of intermetallic compound also increases.
3.4 Porosity

As shown, figure 6 is the porosity in Al matrix composites dispersed intermetallic compound according to the variation of the specific surface area of porous nickel. Porosity in porous nickel, it was occurred by initial polymer foam to fabrication of porous nickel. Polymer foam is three-dimensional network structure like sponge. Therefore, pore inside porous nickel was remained as the hollow struts. When it increases in the specific surface area of porous nickel, pore inside nickel is decreased. In the figure, the pore inside porous nickel decreased by the increasing a specific surface area of porous nickel. Polymer foam can be removed from the metal/polymer by thermal treatment. Although there was almost similar porosity in the matrix infiltrated with porous nickel of three different specific surface area, pore size in the composites infiltrated with high specific surface are is smaller than those with low specific surface area. The general porosity trend in both porous nickel and matrix is reducing with the increasing of the specific surface area at the same fabrication conditions, and the minimum porosity is about 0.17% in the matrix and 0.19% in the porous nickel of >5800 m²/m³ since with the reduction of the porous thickness accelerated the reaction between Ni and Al and delamination caused by the difference of thermal expiation coefficient with nickel, which is a great reduction of defect in the composites as well.

![Fig. 6 Porosity of Al₃Ni, intermetallic compound and Un-reacted nickel.](image)

3.5 Micro-Vickers hardness and Rockwell hardness

Figure 7 shows the results the Micro-Vickers hardness of the Al₃Ni, intermetallic compound and a matrix. The fabrication conditions of composites are applied pressure of 0.1MPa, the 973K temperature of molten Al alloy, holding time of 10 min, and furnace cooling using the porous nickel.

Figure 7 (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 675Hv and 74Hv, respectively. Figure 8 shows the Rockwell hardness of composite by different specific surface area of porous nickel. The Rockwell hardness increasing trend with the increasing of specific surface area. Because amount of intermetallic compound in composite increasing by increasing specific surface area. Rockwell hardness with A scale of composite include intermetallic compound of 28.22% is 45Hₐ. Rockwell hardness of composite include intermetallic compound, 28.22% increased approximately three times compared with matrix. The composite which used over 5800m³/m³ of nickel porous is expected to possess the best potential to improve the mechanical properties of the materials.

5. 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 important results are listed below.
(1) The intermetallic compounds formed by the reaction of porous nickel with molten Al alloy are produced by infiltration-reaction method. Fine and granular intermetallic compound, Al$_3$Ni was observed in the matrix at 973K and 0.1MPa. However, the results indicate that higher the specific surface is, larger the contact surface of porous nickel with molten Al alloy is, and it provided further reaction of Ni with Al.

(2) Number of intermetallic compound, Al$_3$Ni was increasing trend with the increasing of specific surface area. All most of granular-shaped intermetallic compound exists using specific surface area of >5800 m$^2$/m$^3$ and intermetallic compound of 97.5% from an overall rate was observed for aspect ratio of 1.

(3) The general porosity inside composite by using low-pressure infiltration-reaction process method is about 0.36% in the case of using high specific surface area of porous nickel (>5800 m$^2$/m$^3$) since with the reduction of the porous thickness accelerated the reaction between Ni and Al and delamination caused by the difference of thermal expansion coefficient with nickel, which is a great reduction of defect in the composites as well.

(4) The Rockwell hardness increasing trend with the increasing of specific surface area. Because amount of intermetallic compound in composite increasing by increasing specific surface area. Rockwell hardness with A scale of composite include intermetallic compound, 28.22% is 45H$_{RA}$. Rockwell hardness is composite include intermetallic compound, 28.22% increased approximately three times compared with matrix. The composite of high hardness is expected to possess the best potential to improve the mechanical properties of the materials.

Acknowledgements

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References


