Fabrication of Carbon Fiber Oriented Al–Based Composites by Hot Extrusion and Evaluation of Their Thermal Conductivity*1

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New heatsink materials having higher thermal conductivities have recently been required due to the recent rapid improvements in performance of the central processing unit, CPU, with increasing heat generation from computer devices. As an alternative material to conventional heatsink materials such as Al and Cu, composites containing carbon fibers have recently been gaining much attentions because of their extremely high thermal conductivity. However, carbon fiber exhibits high thermal conductivity only in its longitudinal direction. Therefore, it is essential to control the orientation of the carbon fibers in the composite materials. In the present study, hot extrusion of a powder–fiber mixture is applied to realization of unidirectional array of carbon fibers in Al matrix, and the effects of volume fraction of the carbon fibers on the thermal conductivity of the carbon fiber oriented Al–based composite have been investigated. It has been demonstrated that the carbon fibers are unidirectionally oriented in the extrusion direction, and the thermal conductivity in this direction increases with the increase in volume fraction of the carbon fibers. For the composites with more than 30 vol% of carbon fibers, the addition of Al–Si alloy powder or the application of the spark plasma sintering, SPS, before the extrusion was found to be effective for improving the sinterability of the powder–fiber mixture.

Keywords: carbon fiber, aluminum, thermal conductivity, composite, extrusion, orientation control

1. Introduction

There has been a problem with an excessive heat generation in central processing unit due to rapid improvements in performances of electronic devices. Thus, dramatic improvement in heat releasability of heatsink has been strongly required especially for portable electronic devices. The heat releasability of a heatsink placed in the air, Q, can be expressed as eq. (1)1):

\[ Q = \alpha A(T_a - T_i) \]  

where \( \alpha \) is thermal conductivity between the air and the heatsink material, \( A \) is surface area of the heatsink, \( T_a \) is the surface temperature of the fin, and \( T_i \) is the air temperature on the surface. As is clear from eq. (1), it is possible to improve the heat releasability by increasing the surface area of heatsink, \( A \). Therefore, it has been extensively studied how the heatsink design can be optimized for the efficient heat rejection2,3). However, the improvement in the heat releasability of a heatsink by optimizing the device shape has reached a limit in terms of the machinability of the materials4).

Recently, Al-based composites with carbon fibers have been gaining attention as new materials for heatsinks5). Although Al is known to be a light material having a good thermal conductivity, carbon fiber possesses even much higher thermal conductivity than Al, and moreover, it is lighter than Al. Thus, the Al-based composite with carbon fibers can be expected as a new promising super-light heatsink material having a superior thermal conductivity. However, carbon fiber possesses anisotropy in thermal conductivity. In the longitudinal direction it has extremely high thermal conductivity, while in the transverse direction, the thermal conductivity is very low5). Therefore, it is essential to orient carbon fibers unidirectionally to achieve a better thermal conductivity in the Al-based composite. Some methods have been proposed to orient carbon fibers unidirectionally in a metal matrix, for example, pressurized impregnation method6) and preform fabrication by deposition or thermal spray on carbon fibers with subsequent vacuum hot press or rolling7,8). However, these methods still have concerns in orientation and productivity. Thus, in the present study, a hot extrusion process has been focused as a new method which realizes metal forming and carbon fiber orientation simultaneously9). With the hot extrusion process, it can be expected that carbon fibers move with the metal flow through the die hole, and as a consequence, they are oriented in the extrusion direction. In the present study, fabrication of carbon fiber oriented Al-based composite by hot extrusion and investigation of the effect of volume fraction of carbon fibers on the thermal conductivity of the composite have been performed.

2. Experimental

As starting materials of the carbon fiber oriented Al-based composite, commercial Al powder (99.7 mass% purity) and polyacrylonitrile-series carbon fiber, ZY 300 from the Nippon Graphite Fiber Corporation were used. The detailed characteristics of the Al powder and the carbon fiber are described in Table 1. The thermal conductivity of the carbon fiber in longitudinal and transverse directions are 1200 and 10 W m\(^{-1}\) K\(^{-1}\), respectively. The Al powder and carbon fibers were weighed so that the carbon fiber contents should be 0, 2.5, 5.0, 10, 15, 20, 30 and 40 vol% in ideally compacted
composites, and then they were manually mixed together with a spoon in a glass beaker. The mixed powder of 60 g was put into a steel mold, and pressed uniaxially with a load of 340 kN at room temperature to form a cylindrical green compact having a diameter of 45 mm and a height of 15 mm. The compact was placed in a container as shown in Fig. 1, and extruded at 783 K under a load of 340 kN with an extrusion ratio of 20. The extruded bar was columnar with a diameter of 10 mm. The microstructure of the extruded composite was observed with an optical microscope, and phase analysis and thermal conductivity measurement were performed by XRD (X-Ray Diffraction) with CuKα radiation and by a laser flash method(10), respectively. Because there is a concern that the carbon fibers might be broken by severe plastic deformation during extrusion, the fibers were extracted from the extruded composite by immersion in 35 mass% HCl aqueous solution and observed with SEM (Scanning Electron Microscopy). The as-received carbon fibers were also observed for comparison.

3. Results

3.1 Fabrication of carbon fiber oriented Al-based composite

Table 2 exhibits consolidation feasibility of the powder mixture by hot extrusion and relative density of the consolidated composite with respect to the volume fraction of carbon fibers. When the volume fraction is less than 20%, the consolidation is feasible and the relative density is higher than 98%, on the other hand when the volume fraction is more than 30%, the consolidation is not feasible.

Figures 2 (a) and (b) illustrate the outer appearance and longitudinal section of the composite with 20 vol% of carbon fibers. Figure 2 (a) clearly exhibits that the powder mixture was successfully consolidated by hot extrusion. Moreover, in Fig. 2 (b), the carbon fibers are completely oriented in the extrusion direction. Consequently, it has been demonstrated that both the consolidation and the carbon fiber orientation can be realized simultaneously by hot extrusion.

3.2 Thermal conductivity

Figure 3 illustrates thermal conductivities of the extruded composites. The symbols ○ and × indicate the results obtained with the measurements in the longitudinal and transverse directions, respectively. Theoretical thermal conductivities for three different fiber lengths from 20 to 370 μm are

<table>
<thead>
<tr>
<th>Carbon fiber volume fraction (vol%)</th>
<th>Feasibility of extrusion</th>
<th>Relative density (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>+</td>
<td>98.7</td>
</tr>
<tr>
<td>2.5</td>
<td>+</td>
<td>98.7</td>
</tr>
<tr>
<td>5.0</td>
<td>+</td>
<td>98.4</td>
</tr>
<tr>
<td>10</td>
<td>+</td>
<td>98.5</td>
</tr>
<tr>
<td>15</td>
<td>+</td>
<td>98.0</td>
</tr>
<tr>
<td>20</td>
<td>+</td>
<td>98.8</td>
</tr>
<tr>
<td>30</td>
<td>–</td>
<td>NA</td>
</tr>
<tr>
<td>40</td>
<td>–</td>
<td>NA</td>
</tr>
</tbody>
</table>

Table 1 Properties of the starting Al powder and the PAN-based carbon fiber.

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Carbon fiber</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter (μm)</td>
<td>35</td>
<td>10</td>
</tr>
<tr>
<td>Length (μm)</td>
<td>—</td>
<td>370</td>
</tr>
<tr>
<td>Density (g cm⁻³)</td>
<td>2.7</td>
<td>—</td>
</tr>
<tr>
<td>Thermal conductivity (W m⁻¹ K⁻¹)</td>
<td>Longitudinal direction</td>
<td>237</td>
</tr>
<tr>
<td></td>
<td>Transverse direction</td>
<td>10</td>
</tr>
</tbody>
</table>

Fig. 1 Schematic illustrations of (a) extrusion equipment and (b) a die used in the present study.

Fig. 2 (a) The outer appearance and (b) the longitudinal section of the extruded Al-based composite with 20 vol% of carbon fibers.

Fig. 3 Relation between the thermal conductivity of the extruded Al-based composite and the volume fraction of carbon fibers.
also shown in the figure. They were obtained by calculation based on a model\textsuperscript{11} assuming perfect orientation of the fibers in the extrusion direction and taking into account the anisotropy in thermal conductivity of the carbon fiber and the resistance of the thermal conduction at the fiber/matrix interface. The details of the calculation can be found in Appendix.

The measured values in Fig. 3 indicate that as the volume fraction of carbon fibers increases, the thermal conductivity of the composite in the longitudinal direction increases while that in the transverse direction decreases. This is because of the facts that the carbon fibers are oriented in the longitudinal direction of the extruded bar, and that the thermal conductivity of the carbon fibers in the longitudinal direction is larger than that of Al while that in the transverse direction is lower than that of Al.

The calculated results in Fig. 3 indicate that when the fiber length is 370 and when it is 120 μm, the thermal conductivity of the composite in the longitudinal direction increases as the volume fraction of carbon fibers increases. On the contrary, when the fiber length is 20 μm, the thermal conductivity decreases with the increase in the volume fraction. Since the carbon fibers used in the present study have a length of 370 μm, the measured values (circle symbols) should be distributed along the solid line in Fig. 3. However, the circle symbols are distributed far below the solid line, and they are very close to the dot-dashed line showing the calculated results for a fiber length of 120 μm. Possible reason why the measured thermal conductivities were smaller than the calculated ones may be related to (1) formation of a low-thermal-conductivity carbide at the fiber/matrix interface and (2) increment in the resistance of the thermal conduction at the fiber/matrix interface due to increase in area of the interface caused by the fracture of the fibers during the severe deformation of the extrusion. The reason will be discussed in detail in the following sections.

3.2.1 Carbide formation

It has been known that Al\textsubscript{4}C\textsubscript{3} forms at the carbon fiber/Al interface at temperatures above 773 K and it deteriorates the thermal conductivity of composites\textsuperscript{12}. Since the extrusion temperature in the present study was set at 783 K, Al\textsubscript{4}C\textsubscript{3} may have formed in the composite. In order to investigate the existence of this carbide, XRD analysis was performed.

Figure 4 exhibits the XRD analysis results obtained from the composite with 20 vol% of carbon fibers. The detected crystals are only Al, C and Al\textsubscript{2}O\textsubscript{3}, and no peaks for Al\textsubscript{4}C\textsubscript{3} are detected. Thus, it was demonstrated that the reason why the present samples exhibited lower thermal conductivity than theoretically expected values is not due to Al\textsubscript{4}C\textsubscript{3} formation.

The reason for the Al\textsubscript{2}O\textsubscript{3} peak detection can be considered due to the surface oxidation of the starting material of Al powder. This Al\textsubscript{2}O\textsubscript{3} layer cannot be considered to have a significant effect on the thermal conductivity of the composite because the Al\textsubscript{2}O\textsubscript{3} film formed on the Al powder particles should have been broken into tiny pieces during the hot extrusion and newly-born surfaces should be strongly bonded at Al/Al interface.

3.2.2 Fracture of carbon fibers

Figure 5 depicts the carbon fibers before and after the hot extrusion. Although there is no significant difference in surface condition before and after the extrusion, it is obvious that the lengths of carbon fibers were shortened significantly, indicating that the fibers were fractured during the extrusion.

In order to investigate how the fracture of the fiber affects the thermal conductivity of the composite, theoretical values of thermal conductivities were calculated with variety of carbon fiber lengths in a range of 20 to 370 μm (See straight lines in Fig. 3). The effect of the volume fraction of the carbon fibers is strongly affected by the fiber length. The increase in thermal conductivity with increase in the volume fraction of carbon fibers becomes smaller when the fiber length decreases from 370 to 120 μm. Furthermore, when the fiber length is 20 μm, the thermal conductivity decreases with the increase in the volume fraction of carbon fibers, which is a negative effect of the addition of carbon fibers. This is because of the fact that shorter carbon fibers have larger carbon fiber/Al interface area, that is, the higher interfacial thermal resistance exists when the carbon fibers are fractured. Consequently, it has been demonstrated that the reason why the present samples exhibited the lower thermal conductivity than the theoretical values is fracture of the carbon fibers during the extrusion.

The measured thermal conductivity values were very close to the dot-dashed line in Fig. 3, which indicates that the average fiber length in the composite is about 120 μm. Since the original length of the carbon fiber was 370 μm, it can be said...
that the carbon fibers were fractured during the extrusion and the obtained thermal conductivity became much lower than anticipated. As observed in Fig. 5, however, the actual lengths of carbon fibers were less than 120 \textmu m. This point remains to be solved in a future work.

3.3 Sinterability improvement in composites with high carbon fiber contents

In Fig. 3, it has been demonstrated that the thermal conductivity of the carbon fiber oriented Al-based composite improves when it contains high volume fraction of carbon fibers. However, when the volume fraction of carbon fibers was higher than 30\%, the Al powder mixed with the carbon fibers was not successfully extruded into a bar shape and small fragments of the composite were obtained instead, as shown in Fig. 6 (a). In order to find the reason for the failure in consolidation, we observed cross sections of the small fragments and found that open cracks were propagated along the oriented carbon fibers, as shown in Fig. 6 (b). It can be considered that when the volume fraction of the carbon fibers is high, voids are formed between the fibers because the Al powder was too large to fill the voids and it resulted in the crack formation and consolidation failure. That is, the consolidation failure can be considered as a result of increased frequency of carbon fiber agglomeration and void formation associated with the increment in carbon fiber content.

Because higher thermal conductivity can be expected in the composite with higher volume fraction of carbon fibers in general, in order to realize a composite with more than 30 vol\% of carbon fibers, two methods have been proposed in the present study; (1) addition of Al alloy powder having a low melting temperature compared with that of pure Al, and (2) preliminary consolidation before the extrusion.

In the first method, Al-Si alloy powder was added to the mixture of pure Al powder and carbon fibers. The mixture was compacted and extruded at a temperature between the melting temperatures of the pure Al and the Al-Si alloy. During the hot extrusion, the Al-Si alloy liquid can be expected to fill the voids formed between carbon fibers and enhance the sinterability.

In the second method, Spark Plasma Sintering (SPS) was performed for the mixture of the pure Al powder and the carbon fibers prior to the hot extrusion. It was expected that voids in the mixture would be filled before the extrusion due to the high pressure and temperature of SPS, which can result in a good sinterability. In SPS process, pressure from both the upper and lower punches and pulsed great current permeated through the powders are imposed to the sample simultaneously, and the samples are sintered with the discharge plasma generated by the spark discharge phenomenon. Therefore, SPS process can realize a sintering at lower temperatures and at higher speeds differently from other existing sintering methods\(^{13}\). Moreover, since the discharge plasma can remove oxide layers on Al powder surfaces and can lead to the local melting of the Al powder, fully consolidated extrusion billets can be obtained with SPS process.

The above two methods for the sinterability improvement of the composite were verified and the results are discussed below.

3.3.1 Addition of Al-Si alloy powder

As the additive material, Al-12.2 mass\%Si alloy powder with 45 \textmu m in particle diameter was used. The melting temperature of this alloy is 850 K, which is approximately 80 K lower than the melting temperature of the pure Al, 933 K. The alloy powder was added to the mixture of pure Al powder and the carbon fibers of 30 and 40 vol\% and the sinterability was investigated. The content of the alloy powder was set at 10 vol\% in the powder-fiber mixture.

Figures 7 (a) and (c) illustrate the outer appearance and the longitudinal section of the extruded composite bar with carbon fibers of 40 vol\% mixed with the Al-Si alloy powder. The consolidation and sintering were successful even with 40 vol\% of carbon fibers. The relative density of the composites with carbon fibers of 30 and 40 vol\% mixed with the Al-Si alloy powder were 98.0 and 96.0, respectively. From the results, it has been confirmed that the sinterability of the composites was improved by the addition of the Al-Si alloy powder. Figures 7 (b) and (d) illustrate the results obtained with the SPS process, and the results will be discussed in detail subsequently in section 3.3.2. Here, the results of the thermal conductivity measurement of the composite obtained with the Al-Si alloy powder is discussed on ahead.

The symbol * in Fig. 8 depicts the longitudinal thermal conductivity of the composites with the Al-Si alloy powder, while the symbol ◆ depicts the results obtained with the SPS process. The thermal conductivities indicated with the symbol ○ are the same as those in Fig. 3 and are shown here for comparison. The thermal conductivities of the composites with 30 and 40 vol\% of carbon fibers mixed with the Al-Si alloy powder are slightly higher than the results obtained.
from the composite with 20 vol% of carbon fibers without the Al-Si alloy powder. Therefore, the thermal conductivity of the carbon fiber oriented Al-based composite improves as the carbon fiber content increases up to 40 vol%. However, the degree of improvement in thermal conductivity due to the increase in volume fraction of carbon fibers in the composite with the Al-Si alloy powder is lower than that in the composite without the Al-Si alloy powder. In order to investigate the reason why the degree of improvement was deteriorated, the SEM observation and the composition analysis with Electron Probe Micro Analyzer (EPMA) were performed for the composite with the Al-Si alloy powder.

Figure 9 exhibits elemental distributions of Si, Al and C in the composite with 30 vol% of carbon fibers mixed with the Al-Si alloy powder. From Fig. 9 (b), it can be observed that Si exists in between carbon fibers. This result is an evidence of the fact that the Al-Si alloy liquid filled the voids formed in between carbon fibers during the hot extrusion, and as a consequence, the composite was sintered successfully and exhibited a higher thermal conductivity in the longitudinal direction. However, as can be seen in Fig. 9 (b), Si exists not only in between the carbon fibers but also in the Al matrix. The thermal conductivity of Si is 149 W m$^{-1}$ K$^{-1}$, which is much lower than that of Al, 237 W m$^{-1}$ K$^{-1}$. Thus, the existence of Si in the Al matrix deteriorates the thermal conductivity of the matrix11).

Consequently, it can be said that although the sinterability of the mixture of Al powder and carbon fibers can be improved by the addition of the Al-Si alloy powder, Si deteriorates the thermal conductivity of the Al matrix and it results in the deterioration of the degree of thermal conductivity improvement due to the carbon fiber addition.

3.3.2 Preliminary sintering with SPS

The SPS was performed using a graphite die and punch, under a pressure of 10 MPa, at a heating rate of 20 K/min and at a holding temperature of 873 K and for a holding time of 60 min. The mixture of Al powder and carbon fibers was weighed so that the sintered sample became a cylinder of 45 mm in diameter and 17 mm in height.

Figures 7 (b) and (d) depict the outer appearance and the longitudinal section of the extruded composite bar using the SPS-ed billet with 40 vol% of carbon fibers. The sinterability of the composite was improved owing to the sintering before
extrusion, and the composite was able to be extruded into a bar even when the volume fraction of carbon fibers was as much as 40%. The relative densities of the extruded composites with 30 and 40 vol% of carbon fibers were 98.8 and 99.4%, respectively. Therefore, it has been confirmed that the well consolidated composites were successfully fabricated by the SPS followed by hot extrusion. When the volume fraction of carbon fibers is high, the powder-fiber mixture cannot be consolidated only by the extrusion because of the formation of a lot of voids between fibers. However, the SPS can avoid the formation of the voids and make a well-compacted billet that can be extruded into a bar with a high density.

The symbol ◆ in Fig. 8 illustrates the thermal conductivities in the longitudinal direction measured in the composites fabricated with the SPS. The results exhibit higher values than those obtained with the addition of the Al-Si alloy powder (symbol *). Notably, the thermal conductivity of the composite with 40 vol% of carbon fibers fabricated with the SPS exhibits as much as 323 W m\(^{-1}\) K\(^{-1}\), which is 40% higher than that of Al. The degree of thermal conductivity improvement found in the samples fabricated with the SPS is almost the same as that in the samples of lower volume fractions of carbon fibers shown using the symbol ○, and the thermal conductivity increases linearly from 0 to 40 vol% with the increase in the volume fraction of carbon fibers.

In order to investigate whether or not the carbon fibers were damaged by the SPS, the SEM observation was performed on the carbon fibers extracted from the composite fabricated with the SPS followed by the extrusion. The damage level of the carbon fibers was almost the same as that of the carbon fibers extracted from the composite fabricated only by extrusion. Thus, the SPS does not have significant influence on the conditions of the fibers but has strong influence on the density of the compact by reducing the voids between the fibers.

3.4 Conclusions
Fabrication of carbon fiber oriented Al-based composites was attempted and their thermal conductivities were investigated. The results are summarized as follows.
(1) Al-based composite with unidirectionally oriented carbon fibers were successfully obtained by hot extrusion of a mixture of Al powder and carbon fibers. Longitudinal thermal conductivity of the extruded composite bar increases as the volume fraction of carbon fibers increases. When the volume fraction was higher than 30%, the powder-fiber mixture was not extruded into a long bar but into small pieces due to the formation of a lot of voids between fibers.

(2) When Al-Si powder was added, the powder-fiber mixtures with 30 and 40 volume fractions of carbon fibers were successfully extruded into a long bar, because the voids were filled with the eutectic liquid during extrusion. The degree of thermal conductivity improvement due to the increase in the volume fraction of carbon fibers became low because of the low thermal conductivity of Si.

(3) The degree of thermal conductivity improvement increased when the powder-fiber mixtures with 30 and 40 vol% of carbon fibers were well consolidated using SPS prior to the extrusion. The composite with 40 vol% of carbon fibers exhibited 323 W m\(^{-1}\) K\(^{-1}\), which resulted in 40% improvement in thermal conductivity compared with Al.

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Appendix
Here, the explanation will be given regarding a theoretical model which can take into account the anisotropy in thermal conductivity of a carbon fiber and the thermal resistance at the fiber/matrix interface\(^{11}\). A virtual composite unit cell composed of a carbon fiber and matrix is considered and shown in Fig. A1. The thermal conductivities of the cell in x, y and z directions, \(K_x\), \(K_y\) and \(K_z\) are represented in the following eqs. (A1) and (A2). Note that the carbon fiber is assumed to be oriented in z direction.

\[ K_x = K_y = K_m \frac{1 + f \beta_x (1 - L_x)}{1 - f \beta_x L_x} \quad (A1) \]

\[ K_z = K_m \frac{1 + f \beta_z (1 - L_z)}{1 - f \beta_z L_z} \quad (A2) \]

where \(K_m\) is thermal conductivity of the matrix and \(f\) is volume fraction of the carbon fiber. \(\beta_x\), \(\beta_y\) and \(\beta_z\) are expressed in eqs. (A3) and (A4), and \(L_x\), \(L_y\) and \(L_z\) are expressed in eqs. (A5) and (A6).

\[ \beta_x = \beta_y = \frac{K_c - K_m}{K_m + L_x (K_c - K_m)} \quad (A3) \]

\[ \beta_z = \frac{K_c - K_m}{K_m + L_z (K_c - K_m)} \quad (A4) \]
\[ L_x = L_y = \frac{p^2}{2(p^2 - 1)} + \frac{p}{2(p^2 - 1)^2} \cosh^{-1} p \]  
(A5)

\[ L_z = 1 - 2L_x \]  
(A6)

where \( p \) is aspect ratio of the carbon fiber, which is expressed as \( l/d \). Here, \( d \) and \( l \) are diameter and length of the carbon fiber, respectively. \( K^c_x, K^c_y \) and \( K^c_z \) are thermal conductivities of the composite in \( x, y \) and \( z \) directions with the consideration of the interfacial thermal resistance between matrix and the carbon fiber. They are expressed in the following eqs. (A7) and (A8).

\[ K^c_x = K^c_y = \frac{K^c_j}{1 + \frac{2\alpha_k K^c_d}{K^c_m}} \]  
(A7)

\[ K^c_z = \frac{K^c_j}{1 + \frac{2\alpha_k K^c_l}{K^c_m}} \]  
(A8)

where \( K^c_j \) and \( K^c_d \) represent thermal conductivities of the carbon fiber in transverse and longitudinal directions, and their values used in the present study are 10 and 1200 W m\(^{-1}\) K\(^{-1}\), respectively. \( \alpha_k \) in eqs. (A7) and (A8) is called Kapitza radius and expressed as follows.

\[ \alpha_k = R_{bd} K_m \]

where \( R_{bd} \) is a coefficient of the interfacial thermal resistance, which controls the characteristics of interfacial heat transfer. Here \( 8.3 \times 10^{-8} \) m\(^2\) K W\(^{-1}\) was used for the coefficient with reference to the experimental work of Nan\(^{14}\).

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