Influence of Solidification Microstructure and Distribution of Reinforcement on Fatigue Characteristics of Notched SiC Reinforced AC4B Alloy Composites

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The influences of SiC particle distribution, surface notch size and solidification microstructure of the matrix on the fatigue characteristics of SiC reinforced JIS-AC4B alloy composites were investigated. Al–6.79 mass% Si–2.93 mass% Cu–0.17 mass% Mg–0.59 mass% Fe matrix composites with relatively homogeneously dispersed 11 μm SiC particles were fabricated through a combination of pressure infiltration and a melt stirring casting method. The matrix microstructure consisted of a dendritic alpha phase and eutectic Si with a few volume fractions of Fe intermetallic compound among the dendrites. All specimens contained some gas and shrinkage porosities, and all composite specimens contained SiC particle clusters. Vickers hardness of composites clearly increases due to the dispersion of SiC particles and age hardening. The hardening ability increases with an increasing volume fraction of SiC. Rotating bending fatigue tests were carried out on notch-free and notched specimens that had peak aging. In the notch-free matrix alloy specimen, cracks generated from porosities, whereas cracks generated from the SiC particles/the matrix interfaces in the composite specimens. Thus, the fatigue strength decreased with an increase in the SiC volume fraction.

In the notch-introduced matrix alloy specimen, where the stress concentration factor is high, the notch becomes the crack generation site and dominated the fatigue strength. The cracks, however, generate near SiC particles instead of the notch bottom in the composite specimen. Moreover, it was found that the fatigue limit stress is unchanged in composite specimen even when the notch is introduced, although the critical stress for crack generation declines. Microstructural observation revealed that the cracks were spread and diverted in and around the cluster of SiC particles, suggesting that crack propagation resistance was improved in the composite specimen.

In this work, the technique of dispersing SiC particles (from 10 to 30% in volume) in a JIS-AC4B matrix alloy by the combination of pressure infiltration and melt stirring was investigated. Then, the fatigue characteristics of the composites were investigated in relation to the solidification microstructure, the volume fraction and the distribution of particles. Furthermore, we evaluated the influence of an artificially-introduced notch on fatigue crack generation and propagation.

2. Experimental Procedure

SiC particle (Fujimi Incorporated Company Ltd., mean diameter 11 μm, with 0.63 mass% SiO₂ and 0.36 mass% C (hereafter abbreviated as % impurities) was used for the reinforcement and a commercially available Japanese industrial standard AC4B alloy (Al–6.79% Si–2.93% Cu–0.17% Mg–0.49% Mn–0.59% Fe–0.17% Zn–0.03% Sr) was used for the matrix alloy. We combined pressure infiltration and melt-stirring techniques to obtain a suitable volume fraction of SiC particles. The pressure infiltration unit is composed of a metallic mold, a cylinder for pressure and a control unit, and can produce pressures up to 50 MPa, which is high enough to squeeze pores inside the cast composites. The melt-stirring unit is composed of a furnace, a stirring impeller, and a temperature control unit. Flux powder and Ar gas can clean the melt surface and prevent oxidation.

First, AC4B alloy and SiC particles were preheated to 1073 K in a graphite crucible and 873 K in an alumina crucible, respectively. The preheated SiC particles were placed in a cylindrical permanent mold, and then molten AC4B alloy was immediately cast and infiltrated at a pressure of 50 MPa, for 180 s, until solidification completed. The volume fraction of obtained composite block was about 50%. This composite block and AC4B alloy block were remelted in
the melt-stirring unit to attain the specified particle volume fractions to 10 to 30% under 0.2% flux (5MgCl₂ + 3KCl + 2MgF₂) and Ar gas. Stirring was carried out for 3600 s at 60 rpm, on the basis of pre-experimental results, because the particles had a tendency to drop to the bottom with low speed mixing and distribute on the side wall as a result of centrifugal force at high speed mixing. A Sr equivalent about 300 ppm was added into the molten metal for modification of eutectic Si, and this mixture was then stabilized at 935 K for 600 s. Molten alloy with SiC particles were cast into a cylindrical permanent mold and solidified again. Test piece was machined to a 62 mm height, 10 mm thickness and 10 mm width, and performed to solution heat treatment for 36 ks in a salt bath at 773 K and the specimen was then quenched in an ice water bath. Then, the specimens were aged for different times from 30 min (1800 s) to 10⁴ min (6 x 10⁵ s) in a silicon oil bath at 433 K.

The specimens for the tensile strength test and the fatigue test were machined with a lathe from the aged block that achieved the highest hardness during the aging process, as shown in Fig. 1. A sixty degree V form notch with a curvature radius of 0.1 mm and a depth of 0.5 mm was produced on the surface of the fatigue test piece. The bottom surface was polished using silicon carbide, alumina powder, and a diamond paste. An Ono-type rotating bending fatigue tester was used for fatigue testing using a bending moment of 15 Nm, a rotation rate of 50 Hz and a two-way sine wave at a response ratio (R) of −1. The relationship between the cycle number (N) and load stress (σ) that ruptured the specimen was investigated, and the fatigue limit stress was defined as the maximum stress under which no breaking occurred for over 10⁷ cycles. The non-propagating cracks on the surface of the notch bottom and at the cross section of cut specimens were observed using an optical microscope and a scanning electron microscope (SEM). The influences of the introducing notch, the solidification structure and the SiC particle distribution on the fatigue characteristics were investigated.

3. Results and Discussion

3.1 Microstructure observation

The microstructure of the composite material that achieved peak aging at 433 K after the solution treatment is shown in Fig. 2. The SiC particle volume fraction was close to the optimum fraction from the range 10 to 30%, and the particles were distributed relatively homogeneously through the whole specimen. However, clusters of SiC particles ranging from several microns to several hundreds of microns in size were observed. The small cluster sizes result from primary α dendrites growing between the SiC particles, and the large ones may result from insufficient melt agitation. The solidification microstructure of the matrix consists of the primary α phase, the eutectic Si phase and Fe compounds (α-Al₁₁₅(Fe,Mn)₃Si₂ and β-Al₃FeSi). The Fe compound that is in contact with the SiC particle grew a little larger than the other Fe compounds as shown in Fig. 2(c). Blowholes and micro shrinkages of 5 to 150 μm in size were also observed in the specimens. Figure 3 shows the secondary dendrite arm spacing (λ₂) of each specimen. λ₂ became small when the particle volume fraction was increased. In addition, λ₂ was small in the high-density particle distribution within a specimen. Solute diffusion and the dendrite growth direction were restricted to a narrow channel, in comparison to in the unreinforced specimen.

3.2 Age hardening sequence of SiC reinforced composite

The age hardening sequences of the 9.3 vol% and
31.4 vol% specimens at 433 K are shown in Fig. 4. The matrix alloy results are shown for comparison. Matrix alloy is hardened by quenching after solution treatment and then aging at 433 K. The hardness reaches 145 HV after 840 min of aging (peak aging) and the peak hardening response ($\Delta H V$) is 30 HV. Similarly, an increase in the hardness is observed even in the aging 9.3 vol% composite materials. The hardening is caused by the SiC particles themselves and the dislocations introduced at the time of quenching, due to the thermal expansion coefficient difference between the particle and matrix, as reported for Al$_2$O$_3$/AC4B$_{13-15}$ and SiC/Al alloy$_{16,17}$ composites. Although the 9.3 vol% composite hardens with aging, peak aging is achieved in a shorter period of time, 780 min. A similar result was obtained for the 31 vol% composite; a peak hardness of 220 HV was reached in 660 min. These results are summarized in Fig. 5. The aging response and the peak aging hardness increase with an increasing SiC volume fraction, but the time required to attain peak aging decreases. This is because the dislocations introduced in the vicinity of the SiC particles promotes the precipitation of phases such as the $\lambda'$ (Al$_3$Cu$_2$Mg$_8$Si$_6$) and the $\theta'$ (Al$_2$Cu) that contribute to aging.$^{13}$ In addition, as shown in Fig. 4(b), the difference of microhardness in the dendrite center is not influenced as much by SiC, which indicates that the dislocations had no influence at large distances from the SiC particles. The specimen with the highest macro hardness was used for the tensile strength test and the fatigue test.

### 3.3 Influence of particle distribution on tensile strength

The mechanical properties of each specimen are shown in Table 1. The tensile strength, 0.2% yield strength and elongation of the composite specimens are lower than those of the matrix alloy, and they decrease with an increase in the SiC volume fraction. The minute cracks that generate at eutectic Si and Fe compounds are observed in great numbers, and these cracks occur from a broken surface to a width of about 5 mm in the matrix alloy. This means that plastic deformation occurs over a comparatively wide area before the matrix specimens rupture. On the other hand, in the composite specimens, a few larger cracks were observed.
around SiC particles in the local area range of about 1 mm
from the broken surface. Therefore, a cluster of SiC particles
may concentrate stress, and immediately cause a crack and
rupture.

3.4 Influence of particle distribution on fatigue characteristics of notch-free specimen

The results of the fatigue tests on the matrix alloy
specimen and the 9 vol% and 31 vol% SiC specimens are
shown in Fig. 6. The arrows in the figure show that the specimen did not rupture
even with a number exceeding $10^7$ cycles, so experiment stopped.

Fig. 6 Relationship between applied stress and number of cycles in the
rotating bending fatigue test for notch-free composites and matrix alloy
specimens. The arrows in the figure show that the specimen did not rupture
even with a number exceeding $10^7$ cycles, so experiment stopped.

Table 1 Mechanical properties of matrix AC4B alloy and composite specimens.

<table>
<thead>
<tr>
<th></th>
<th>tensile strength $\sigma_t$ (MPa)</th>
<th>yield strength $\sigma_{0.2}$ (MPa)</th>
<th>elongation $\epsilon$ (%)</th>
<th>Vickers hardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>matrix alloy</td>
<td>345.2</td>
<td>287.0</td>
<td>1.58</td>
<td>140</td>
</tr>
<tr>
<td>9.3 vol% SiC composites</td>
<td>315.2</td>
<td>279.1</td>
<td>0.94</td>
<td>150</td>
</tr>
<tr>
<td>31.4 vol% SiC composites</td>
<td>299.3</td>
<td>268.9</td>
<td>0.69</td>
<td>155</td>
</tr>
</tbody>
</table>

Table 2 The amount and size of the porosity in matrix AC4B alloy and composite specimens.

<table>
<thead>
<tr>
<th></th>
<th>maximum diameter ($\mu$m)</th>
<th>average diameter ($\mu$m)</th>
<th>number of particles ($/\text{mm}^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>matrix alloy</td>
<td>25.0</td>
<td>4.5</td>
<td>23</td>
</tr>
<tr>
<td>9.3 vol% SiC composites</td>
<td>32.6</td>
<td>9.9</td>
<td>26</td>
</tr>
<tr>
<td>31.4 vol% SiC composites</td>
<td>48.4</td>
<td>12.7</td>
<td>41</td>
</tr>
</tbody>
</table>

and 120 MPa for the 9 vol% and 31 vol% specimens, respectively. These values are lower than that of the matrix
alloy and decrease with an increase in the SiC volume fraction. These results were also scattered, even though the
specimens were machined from a single piece of composite block. It is generally recognized that experimental results for
fatigue tests on notch-free specimens are significantly scattered, since there are differences in stress distribution
and concentration in specimens even at the same nominal stresses, due to different internal cast defects, sizes, shapes
and positions. Therefore, the amount and size of the porosity were investigated, and both parameters were found
to be higher in the composites, as shown in Table 2. Hence, the increase in the volume fraction of porosity could be one
of the reasons for the decline in the fatigue strength.

However, a large amount of the fine non-propagating
cracks from the clusters of SiC particles were observed at the
specimen surface. The Young’s moduli of the matrix alloy
and the SiC particle are about 72 GPa and 560 GPa, so the
matrix alloy causes plastic deformation at the SiC particle/
matrix interface. Thus, the broken sections of the test pieces
were analyzed by a scanning electron microscope. It was seen
that cracks generate from several points on the specimen
surface, and then progress radially toward the insides of the
specimens, as shown by the arrows in Figs. 7(a) and 8(a).
Moreover, clusters of SiC particle were observed where the
cracks generated (Fig. 7(b), Fig. 8(b)). SiC particle clusters
from 80 to 190 $\mu$m in size were seen in the 31 vol% specimens corresponding to the data in Fig. 6. Accordingly,
it is concluded that the cracks generate at the interfaces
between SiC particles and the $\alpha$-dendrite, and that the clusters of SiC particles, rather than the porosity control fatigue crack
generation in the composite materials. Furthermore, the
fatigue results depend on the distribution of the size of SiC
particle clusters inside each specimen (Fig. 6). Toda et al.
conclude that the higher aspect ratio or sharp corner of the
reinforcement results in the decreasing microscopic strain, and Llorca et al. suggest that high volume fraction area has a
tendency to become embrittled. Thus, the crack can easily
initiate vicinity of the SiC particle cluster, suggesting that
fatigue limit stress can be increased if the SiC particles are
distributed homogeneously and the cluster size is controlled
to be small. In addition, since an uneven fracture surface is
Fig. 7 Scanning electron micrographs of fractured surfaces for 9.3 vol% SiC/AC4B composites in applied load of 130 MPa (a). There are clusters of SiC particle (arrows in (a) and (b)), uneven fracture surface (c) and striation pattern (d) on the surface.

Fig. 8 Scanning electron micrographs of fractured surfaces for 31.4 vol% SiC/AC4B composites in applied load of 120 MPa.
observed, as shown in Figs. 7(c) and 8(c), the destruction of the test piece might result from the repetition of the crack propagating step and the connection of separate cracks. The width of each striation pattern as shown in Figs. 7(d) and 8(d) suggests that the crack propagating step of the 9 vol% SiC specimen after 1 cycle is smaller than that of the 31 vol% specimen under the a similar applied stress of 120–130 MPa.

3.5 Influence of the notch on fatigue characteristics

Figure 9 shows the S–N curve obtained for the 0.1 mm notched specimens. The cycle number (N) increases with a decline in the setting load, similarly to the case for the notch-free matrix alloy, and the notch fatigue strength was found to be σ = 65 MPa. On the other hand, the fatigue strengths of the composites become 95 MPa and 105 MPa for the 9 vol% and 31 vol% SiC specimens, respectively. These values are higher than that for the matrix alloy. Furthermore, the fatigue limit strength increases as the SiC volume fraction increases. Thus, the macrostructure of the specimen that did not rupture even at N = 10⁸ was observed. In the matrix alloy, cracks progressed only on the base surface of the notch. However, in the composite specimens, cracks were observed over the entire circumference, zigzagging over a relatively wide width range of about 5 mm, centering on the notch. The cracks progressed in the interface between the SiC particle and the α-dendrite, or in the vicinity of the SiC particles. In particular, many fine non-propagating cracks were intertwisting, branching and deflecting in a complicated manner in the presence of clusters of SiC particles in the 31 vol% SiC specimen. The fatigue fracture surfaces about the 0.1 mm notches for the 9 vol% and 31 vol% SiC specimens, as observed by SEM, are shown in Figs. 10 and 11. Many cracks occur on the surfaces of the specimens especially around the notch, and then the cracks progress radially toward the centers and inside of the specimens to obtain a complicated fracture surface, similarly to that for the notch-free composites. As shown in Figs. 10(b) and 11(b), clusters of SiC particles are observed where the cracks are generated, and an uneven fracture surface is observed around the cluster of SiC particles as shown in Figs. 10(c) and 11(c). The uneven fracture surface is as large as the dendrites, and smaller than in the matrix alloy. Striation pattern was also observed, as shown in Figs. 10(d) and 11(d). The progression of the crack was deflected by the SiC particle clusters. From the aforementioned observation, it can be concluded that, although SiC particle clusters work as generation points for cracks, crack propagation is sometime halted in SiC particle clusters, and then the concentration of stress in each crack is mitigated, so that the many discrete cracks do not contribute to the destruction of the specimen.

3.6 Influence of stress concentration factor on fatigue limit stress

The relationship between the applied stress (σ) and the stress concentration factor (Kt) defined by the notch depth (w) and radius (r) is shown in Fig. 12. Since some specimens did not break even if there was a crack, the crack generation stress (σw1), where cracks are initiated, and the fatigue limit stress (σw2), where cracks start to propagate to contribute for the fracture formation, were evaluated. σw1 is determined by investigating the outside of a test piece in detail, and σw2 is determined as the fatigue limit stress from the S–N curves of Figs. 6 and 9. The highest fatigue limit stress was 140 MPa for the matrix alloy, while the fatigue stresses were 110–120 MPa for the composites at a Kt of 1 (notch-free specimen). No evident difference between σw1 and σw2 was observed. On the other hand, with a notch radius of 0.1 mm, where Kt = 3.76, the σw1 values of all specimens decreased remarkably, to 55 MPa. Cracks are introduced forcibly by the existence of the notch, and the generation of cracks is not influenced by the distribution of SiC particles. However, σw2 is unchanged with increasing Kt, and values of 95 MPa and 105 MPa are obtained for the 9 and 31 vol% SiC composite specimens, although σw2 decreases in the matrix specimen. The fatigue strength of 105 MPa for the 31 vol% SiC specimen is almost equal to the value of 110 MPa for the notch-free specimen.

The σw1 and σw2 values for each specimen are summarized in Fig. 13 in relation to the particle volume fraction. σw2 increases with an increasing SiC volume fraction, and the range, over which the specimen does not break even if cracks are generated, was expanded. Thus, particle distribution would influence the crack spread, progress, connection and so on. The notch sensitivity of each specimen was evaluated on the basis of linear notch mechanics. Figure 14 shows the relationship between σw1, σw2, and σw0 (the fatigue strength of notch-free material) and Kt. The Kσw2/σw0 value for unreinforced alloy is almost unchanged at 1 to 1.5, indicating that σw2 lowers inversely proportional to the increasing of stress concentration. However, Kσw2/σw0 increases to 3.5 for 31% SiC specimen, suggesting that σw2 improves regardless of the stress concentration and that composite specimen has the lower notch sensitivity.

The microstructure of the fatigue fracture surface in the unreinforced specimen at a 45-degree angle is shown in Fig. 15(a). The crack in the matrix alloy progressed in a
Fig. 10 Scanning electron micrographs of fractured surfaces for notched 9.3 vol%SiC/AC4B composites. The applied load is 100 MPa, and notch radius is 0.1 mm.

Fig. 11 Scanning electron micrographs of fractured surfaces for notched 31.4 vol%SiC/AC4B composites. The applied load is 110 MPa, and notch radius is 0.1 mm.
straight line along the notch (Fig. 15(b)). In contrast, in the composites, the many fatigue cracks occur to a large extent everywhere, and form intense complicated fractures as shown in Fig. 15(c, d). This tendency is clearer in the 31 vol% SiC specimen; 30% and 70% of the cracks generate from places other than the notch in the 9 and 31 vol% SiC specimens, respectively. In these specimens, the stress concentration might be mitigated since the crack tip is branched and deflected by SiC particle clusters. The crack progress might also decrease because the rough crack surfaces bump into each other. Toda et al. developed the theory when the crack progress into ceramics particle reinforced alloy, and pointed out that the micro-crack, stress concentration, deposition of reinforcement from matrix and plastic deformation of matrix changed the crack propagating direction. Thus, similar crack branching occurred in present condition. Furthermore, the secondary dendrite arm is shortened and the intermetallic compound densely scattered for high volume fraction composite because of the obstruction of SiC particles as shown in Fig. 3. Finer grain may also enhance the crack branching. Thus, it can be concluded that, since the frequency of inflection and branching of the crack increases with an increasing SiC particle volume fraction, crack propagation resistance and eventually $\sigma_{w2}$ are improved.

4. Conclusions

The influence of reinforcement on dendrite growth and fatigue characteristics of a metal matrix were investigated for notched SiC particle/AC4B (Al–Cu–Si–Mg) alloy composites. The results obtained are summarized as follows:

(1) By combining pressure infiltration and melt stirring processes, it was possible to disperse SiC particles of specific volume fractions comparatively uniformly.

(2) The composite materials show obvious age hardening, and age hardening ability increases with an increasing SiC volume fraction.

(3) For unreinforced specimens, the rotating bending fatigue limit stress is influenced the notch size, and the stress decreases as notch size decreases.

(4) The SiC reinforced specimens show the same fatigue sequence in smooth and notched surface specimens, indicating that the fatigue strength is determined by the distribution of SiC particle clusters.
(5) The clusters of SiC particles may be one of the reasons for the increase in the plastic deformation resistance against crack propagation, due to the extended crack length and the branching in crack growth directions, and eventually improve the fatigue characteristics.

REFERENCES


Fig. 15 Scanning electron micrographs of the fatigue fracture surfaces for unreinforced AC4B alloy (a) and 31.4 vol% SiC reinforced composites (c) at a 45-degree angle, and schematic illustrations of fracture mechanisms in the unreinforced alloys (b) and composites (d).