In-situ Observations of Fracture Processes in 0.6 μm and 9.5 μm SiCp/6061Al Composites

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In-situ SEM observations of fracture processes in two 6061Al alloy composites reinforced with coarse and fine SiC particles, respectively, were carried out to clarify their fracture mechanisms. It was found that in the coarse particle reinforced composite, voids were formed in the matrix around SiC particles ahead of the main crack tip, then coalesced with each other, and finally connected with the main crack tip, causing propagation of the main crack. However, in the fine particle reinforced composite, multiple micro-cracks were formed at the boundaries between SiC particle clusters and surrounding matrix or within the clusters, then connected with each other, and finally joined with the main crack tip, leading to crack branching and growth of the main crack. Crack branching, multiple cracking and crack deflection were proposed to contribute to the enhanced fracture toughness in the fine particle reinforced composite compared with the coarse particle reinforced composite.

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1. Introduction

Addition of discontinuous silicon carbide particles to the aluminum alloys can appreciably increase the specific elastic modulus and specific strength at both room and elevated temperatures. However, the ductility and fracture toughness of these composite materials are drastically degraded compared with those of their matrix alloys.1–4) The degradation in fracture toughness is influenced by many factors, such as particle size, aspect ratio, volume fraction, distribution and strength of the particles, strength of the interface between the particles and matrix, and properties of their matrix alloys.5–8)

It has been recognized that an increase in volume fraction of SiCp leads to a decrease in fracture toughness of the composite,5–7) but an increase in SiC particle size causes an increase in fracture toughness.8) In the previously reported work,3,5–8) the studied SiC particle sizes were larger than 2 μm. More recently, however, a result in our research5) indicated that the fracture toughness (both crack initiation toughness and propagation toughness) of the composite reinforced with much finer SiC particles (0.6 μm) was obviously higher than that of the composite reinforced with coarse particles (9.5 μm) although the volume fraction of the reinforcements was kept the same. The present work is an extension of Ref. 4, attempting to clear up the mechanism for the enhanced fracture toughness in the finer particle reinforced composite.

To understand the fracture mechanisms and to rationalize the degraded fracture toughness of the Al alloy matrix composites, investigators have inspected the fracture surfaces and cross sectional microstructures beneath close to the fracture surfaces by using SEM after fracturing the specimens.3–9) They found that the fracture modes in the SiCrp/Al alloy composites mainly included the failure of their matrix, the fracture of interfaces between the particles and matrix, and the fracture of particles themselves. All their inspections, however, were made on the post-fractured specimens, therefore unable to disclose the detailed fracture processes during loading.

In addition, how to determine and detect the crack initiation point is always a tough problem. Although the direct current electrical potential technique and the acoustic emission method can be used to estimate the crack initiation in the case of static loading, they are not feasible for dynamic loading. The compliance change rate method developed by Tseng et al.10) and Kobayashi11,12) has been successfully applied to steel, iron, titanium alloys and aluminum alloys.12,13) In the compliance change rate method, the abrupt change point of the compliance change rate is assumed to be the crack initiation point, which corresponds to the formation of a critical stretched zone width at the crack tip in the tested specimen. However, the critical stretched zone width at the crack tip in composite materials is difficult to detect. Therefore, the practical meaning of the abrupt change point of the compliance change rate in particle reinforced composites needs to be clarified.

In this paper, in-situ SEM observations of the crack evolution were made on two 6061Al matrix composites respectively reinforced with fine and coarse SiC particles of the same volume fraction. The purpose of the present research is, by extending our previous work,5) to provide a complete picture of the fracture processes in the composites and make a clear understanding of (1) the fracture mechanisms and the difference in fracture mechanisms of the two particle reinforced composites, and (2) the physical meaning of the abrupt change point of the compliance change rate in the composite materials.

2. Experimental

The employed two composites were 6061Al alloy reinforced with 15 vol% SiC particles of 9.5 μm and 0.6 μm in size, respectively. These composites were fabricated by a conventional method of vortex melting followed by squeeze casting.14) First, molten 6061Al alloy at 750 °C was stirred and mixed with preheated SiC particles at a stirring speed of
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3. Results

3.1 Fracture processes in the 9.5 μm SiC_{p}/6061Al composite

The bending load-displacement curve of the 9.5 μm SiC_{p}/6061Al composite is shown in Fig. 1. The corresponding compliance change rate curve is also plotted in Fig. 1. The compliance change rate, \( \Delta C/C \), is defined as

\[
\Delta C/C = (C - C_{el})/C_{el}
\]

where \( C = d/P \) is the compliance of any point on a load-displacement curve, i.e., the linear compliance from the zero load point to any reached load point, \( P \) and \( d \) are the corresponding load and displacement at the reached point, respectively, \( C_{el} = d_{el}/P_{el} \) is the compliance of linear elastic section on the load-displacement curve, and \( P_{el} \) and \( d_{el} \) are the elastic limit load and the displacement at the elastic limit load, respectively.

The successive micrographs taken during bending are shown in Figs. 2(a)–(h). These graphs correspond to the loading points alphabetically indicated in Fig. 1. As load was applied, the pre-fatigued crack tip was gradually opened and a severe deformation zone (as illustrated by the white band in Fig. 2(a)) appeared around the crack tip and at both sides of the crack. During the whole elastic deformation process, cracking of SiC particles or debonding between the particles and surrounding matrix were hardly found.

When the specimen was loaded to plastic deformation region (point b in Fig. 1), the white deformation band at and behind the crack tip was widened, the crack tip was further opened (Fig. 2(b)) and a detectable crack growth could be seen. At loading point c in Fig. 1, the crack grew by a distance of about 1.5 μm and the opening of the original crack tip (OCT) was increased by 2.5 μm, as shown in Fig. 2(c). As the load was increased to point d in Fig. 1, which corresponds to the abrupt change point on the compliance change rate curve, the original crack tip opening displacement (OCTOD) was \( \sim 8.2 \) μm, the crack extension was \( \sim 3.5 \) μm and the severe deformation zone (SDZ) in front of the crack tip was \( \sim 30 \) μm. The severe deformation zone at the moment covered several SiC particles ahead of the crack tip, however there were no cracking of SiC particles and formation of voids around the particles (Fig. 2(d)).

When the load was increased to its maximum (point e in Fig. 1), the OCTOD reached \( \sim 18 \) μm and the crack grew by \( \sim 12 \) μm (Fig. 2(e)). At this loading point, the SDZ size in front of the crack tip was \( \sim 45 \) μm, covering more SiC particles ahead of the crack tip than in Fig. 2(d). Clearly, voids were formed in the matrix around a large SiC particle nearest to the crack tip and also in the matrix around several other SiC particles in front of the crack tip as indicated by arrows.

When the load reached point f in Fig. 1, the OCTOD was further increased and the crack grew more (Fig. 2(f)). The SDZ was enlarged rapidly to a size of 75 μm. New voids were formed and the growth of previous voids was found in the deformation zone (as indicated by arrows). During the subsequent loading, more new voids were initiated in a distance in front of the crack tip and the voids near the tip were enlarged. The voids were coalesced with each other and connected with the main crack, leading to the propagation of the main crack (Figs. 2(g) and (h)).

3.2 Fracture processes in the 0.6 μm SiC_{p}/6061Al composite

The load-displacement curve and the compliance change rate curve of the 0.6 μm SiC_{p}/6061Al composite during
Fig. 2 Sequential micrographs taken in the 9.5 µm SiCp/6061Al composite during loading. Different graphs correspond to the loading points alphabetically shown in Fig. 1.
bending are plotted in Fig. 3. The micrographs sequentively taken during bending (C) are shown in Figs. 4(a)–(i). The loading points corresponding to these photographs are alphabetically designated in Fig. 3. At the lower loading point a in Fig. 3, the profile of the fatigue crack tip and the microstructure around the crack tip are shown in Fig. 4(a). It is noted that the fine SiC particles were distributed nonuniformly in the form of cluster colonies. After slightly increasing the load to point b in Fig. 3, a large crack branching (B1 in Fig. 4(b), which could have been noticed but couldn’t have been seen clearly in Fig. 4(a)) was clearly seen around the main crack tip, and a sub-branching was on the large branching (B11 in Fig. 4(b)). Some other small branches were observed at other sites in the area around the crack tip (e.g. B2 and B3 in Fig. 4(b)). These crack branches were formed by the pre-fatigue test before bending and were located in the matrix beside or inside SiCP clusters. The main crack was slightly opened and a severe deformation zone became visible around the crack (highlighted by white color).

When the specimen was loaded to the plastic deformation region (point c in Fig. 3), a void emerged around a small cluster composed of fine SiC particles about 3 μm away from the main crack tip. Two small micro-cracks appeared. One micro-crack (C1 in Fig. 4(c)) was about 12 μm away and the other (C2 not shown in Fig. 4(c)) was about 30 μm away from the front of the main crack tip (i.e., at the boundary between a SiCP cluster and its surrounding matrix and between two SiCP clusters, respectively). The original crack tip (OCT) was opened by ~1.4 μm and the crack extended by ~1 μm (Fig. 4(c)). In addition, the severe deformation zone around the crack was widened.

When the load was increased to point d in Fig. 3, the two micro-cracks C1 and C2 became longer, one 5 μm long and the other 3 μm long (Fig. 4(d)). The OCT was opened by 2 μm and the crack extension increased by 1.6 μm. When the load was further increased, the two small micro-cracks grew larger and a plastic deformation band in a fan shape occurred in the matrix ahead of the two micro-cracks (Fig. 4(e)).

When the load was increased to point f (Fig. 3), which corresponded to the abrupt change point on the compliance change rate curve, the two micro-cracks C1 and C2 joined each other, and connected with the main crack (i.e. B4 in Fig. 4(f)). In addition, a new micro-crack was formed inside a SiCP cluster in close front of the crack tip as indicated by C3 in Fig. 4(f). The original crack tip opening displacement increased by ~4 μm. The deformation zone in front of the main crack tip extended for ~54 μm.

After further increasing the load, the CTOD increased and the formerly formed two micro-cracks C1 and C2 completely joined with the main crack. The newly formed micro-crack C3 was enlarged, several other small cracks appeared close to C3, and another new micro-crack C4 was formed in front of the micro-crack C3 at the boundary between the same SiCP cluster and matrix (as indicated in the middle lower part in Fig. 4(g)). During the subsequent loading, the crack earlier formed by the connection of two micro-cracks with the main crack (B4 in Fig. 4(g)) stopped growing, while the newly formed micro-cracks C3 and C4 grew rapidly along the boundary between the SiCP cluster and matrix.

When the load was increased to the maximum (point h in Fig. 3), the new micro-cracks connected and linked to the main crack (Fig. 4(h)). The length of the connected micro-cracks reached to a size of about 40 μm. The original crack tip opening displacement was around 14 μm and the plastic deformation band in front of the main crack covered a size of 100 μm. With further loading, the main crack propagated along the newly formed crack and along the deformation band (Figs. 4(h) and (i)) until final failure, whereas the previously formed crack (B4 in Figs. 4(h) and (i)) acted as large branching.

4. Discussion

4.1 Fracture mechanism

In a model proposed by Rice and Johnson and developed by Refs. 17 and 18, fracture toughness can be predicted by the crack tip opening displacement if fracture of a material is characterized with dimple morphology,

\[ K_{IC} = \frac{E\sigma_0\delta}{\alpha(1-\nu^2)} \]  

and

\[ J_{IC} = \sigma_0\delta/\alpha \]  

where \( K_{IC} \) and \( J_{IC} \) are the critical fracture toughness and critical \( J \) integral, respectively, \( \delta \) is the crack tip opening displacement (CTOD) in the material at the time when crack begins to propagate, \( \sigma_0 \), \( E \) and \( \nu \) are the yield strength, young’s modulus and Poisson’s ratio, respectively, and \( \alpha \) is a proportional factor depending mainly on the strain-hardening exponent, \( n \), and stress states. From this proposition, it can be deduced that the wider the CTOD when crack begins to grow, the higher the fracture toughness of the material.

It has been identified that the fracture surfaces of SiC particle reinforced composites showed dimple morphology. The CTODs (\( \delta \)) measured for the two composites at the abrupt change points of compliance change rate (ACPs) and at the maximum loading points are summarized in Table 1. At the ACPs, the CTODs are about 8 and 4 μm for the composite reinforced with coarse SiC particles and for the composite reinforced with fine SiC particles, respectively.
Fig. 4 Sequential micrographs taken in the 0.6\( \mu \)m SiC\(_p\)/6061Al composite during loading. Different graphs correspond to the loading points alphabetically shown in Fig. 3.
composite reinforced with fine SiC particles, respectively. Alternatively, at the maximum loading points, the CTODs of the two composites are about 18 and 14 μm, respectively. Thus, according to Rice and Johnson’s model, higher crack initiation toughness should be expected for the coarse particle reinforced composite when the crack initiation point is determined by either the compliance change rate or the maximum loading point method. The yield strength of the coarse and fine particle reinforced composites is 153 and 152 MPa, respectively. The parameter α has a value of 0.33 for the present composite materials with a strain-hardening exponent n = 0.2 from Shih’s report. The crack initiation toughness predicted according to eq. (3) is 3.8 and 1.8 kJ m⁻² based on the CTODs at the ACPs, and is 8.3 and 6.4 kJ m⁻² based on the CTODs at the maximum loading points, respectively for the coarse and fine particle reinforced composites as tabulated in Table 1. In our recent reports, however, the fracture toughness measured according to the ASTM standard method is lower for the coarse particle reinforced composite than that for the fine particle reinforced composite (Table 1). The measured crack initiation toughness is 4.9 and 7.0 kJ m⁻² when the crack initiation point is determined by the ACP, and is 8.7 and 11.3 kJ m⁻² when the crack initiation point is determined by the maximum loading point, respectively for the coarse and fine particle reinforced composites. Apparently, for the composite reinforced with coarse particles, the fracture toughness predicted according to Rice and Johnson’s model based on either the ACP or the maximum loading point are comparable to the measured values. However, for the composite reinforced with fine particles, the predictions based on both the ACP and the maximum loading point are much lower than the measured values. The actual enhanced fracture toughness in the fine particle reinforced composite when compared with the coarse particle reinforced composite, and the disagreement between the predictions and the measurements, are explained as follows.

At first, it should be pointed out that the nature for the enhancement in fracture toughness in the fine particle reinforced composite is not due to the smallness of particle size but associated with the formation of particle clusters. To some extent, a cluster of fine particles might be thought of as a big particle, i.e. the “effective” size of the reinforcements in the clustered composites could be of the size of clusters. The average cluster size in the fine particle composite was quantified to be ~18 μm from the SEM micrographs.

Accordingly, the “effective” particle size of the fine particle composite was assumed to be of this order. In this manner, the improved fracture toughness in the fine particle composite seems accountable, and appears in agreement with the concept that an increase in particle size gives rise to an increase in fracture toughness. However, from the CTOD point of view (eq. (4)), the smaller CTOD couldn’t explain the higher fracture toughness in the fine particle composite. In nature, the toughening mechanisms in the clustered fine particle composite are not that simple and different from those in the coarse particle reinforced composite according to the following analysis.

To help understand the facture mechanisms in the two composites, SEM fractographs of the crack initiation zones ahead of the fatigue pre-crack tips were taken and two typical micrographs are shown in Figs. 5(a) and (b) respectively for the coarse and fine particle reinforced composites. In the coarse particle composite, dimples of size of ~10 μm are seen and assumed to be related to the reinforcing SiC particles as confirmed by some particles contained in the dimples. For the fine particle composite, a lot of zones of large sizes of 15~20 μm are obvious, which are assumed associated with fractured clusters. In each of these zones are seen a large number of small dimples, which are due to the fine SiC particles. In the bottom of these zones, occasionally

**Table 1** Crack tip opening displacements (CTODs) measured at the abrupt change point of compliance change rate and the maximum loading point, the predicted fracture toughness (\(J_{IC,P}\)) and the measured fracture toughness (\(J_{IC-M}\)).

<table>
<thead>
<tr>
<th>Crack initiation point of SiC/6061Al</th>
<th>CTOD, (\delta/\mu m)</th>
<th>(J_{IC,P}), kJ m⁻²</th>
<th>(J_{IC-M}), kJ m⁻²</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACP</td>
<td>Coarse SiC</td>
<td>8.2</td>
<td>3.8</td>
</tr>
<tr>
<td>MLP</td>
<td>Coarse SiC</td>
<td>18</td>
<td>8.3</td>
</tr>
<tr>
<td></td>
<td>Fine SiC</td>
<td>4</td>
<td>1.8</td>
</tr>
<tr>
<td></td>
<td>Fine SiC</td>
<td>14</td>
<td>6.4</td>
</tr>
</tbody>
</table>

Note: ACP-The abrupt change point of compliance change rate
MLP-The maximum loading point

*Fig. 5* Typical fractographs of the crack initiation zones for the coarse (a) and fine (b) particle reinforced composites. The arrow indicates a secondary crack.
seen are secondary cracks or micro-cracks as indicated by an arrow in Fig. 5(b). The obvious differences in fracture surfaces of the two composites indicate their different fracture mechanisms. Based on the in-situ observations of fracture processes and SEM micrographs of fracture surfaces, it becomes clear that the crack initiation of the coarse particle reinforced composite was mainly associated with the formation of voids in the matrix around individual particles ahead of the main crack tip. The crack propagation and fracture of the composite was caused by coalescence of the voids and their connection with the main crack. On the contrary, the boundaries between the particle clusters and their surrounding matrix, and the matrix within the clusters were responsible for the crack initiation and failure of the fine particle reinforced composite.

A schematic illustration of the microstructural evolution around the fatigue pre-cracks during loading is shown in Fig. 6. In the case of coarse particle reinforced composite, slight crack deflection is formed in the wake zone behind the fatigued crack tip. When a small load is applied, the crack at the tip and in the wake zone is almost fully opened. With increasing load, voids are separately initiated around individual SiC particles and coalescence of these voids causes the crack propagation. Whereas in the case of fine particle reinforced composite, crack branches are seen in the wake zone behind the fatigue pre-crack tip. When the applied load is small, crack faces are almost contacted with each other. With further loading, multiple micro-cracks are initiated around or within clusters because multiple particles are existent as crack initiation points and additional internal stress elevation occurs within each cluster. Consequently, new crack branching is generated before the main fatigue pre-crack is propagated.

The enhanced fracture toughness in the fine particle reinforced composite is assumed to be associated with several factors. The most influential factors concerned here are crack branching, micro-cracking and crack deflection. The toughening contribution of crack branching comes from the two aspects: energy consumption and crack tip shielding. Firstly, energy consumption is needed for the formation of new crack branching since crack branching yields an increase in the crack surface; secondly, crack branching in the wake zone shields the crack tip from the far-field applied stress intensity by decreasing the CTOD, hence, the effective stress intensity, which actually acts on the crack tip, is decreased.

Multiple micro-cracks were extensively observed around or within fine SiC particle clusters. In our previous work, parametric studies were performed on the effect of micro-cracks on the crack initiation and fracture behavior in a 6061Al matrix composite reinforced with clustered SiC whiskers. Those studies analyzed the crack driving force in the presence of micro cracking near a main crack tip, and indicated that mixed mode (modes I and II) stress intensity factor existed. The mode II stress intensity factor fluctuated significantly when the main crack encountered multiple micro-cracks where reinforcement agglomeration existed, which resulted in a remarkable crack deflection to the clustered reinforcements. On the other hand, when the micro-cracks were located behind or at a small distance ahead of the main crack tip, the mode I stress intensity factor was reduced drastically due to the anti-shielding and shielding effects caused by the clustered micro-cracks, thus, the extension of the main crack was strongly retarded. Lastly, the deflection of the main crack unavoidably increased the real fracture surface, further enhancing the fracture resistance. Hence, the fracture toughness in terms of critical far-field stress intensity factor in the fine particle reinforced composite was enhanced when compared with the composite reinforced with coarse particles in the present study.

The above analysis suggests that the toughening mechanisms of particle clusters and large particles in composites are different. Crack branching formed between SiC particle clusters and their surrounding matrix, shielding and anti-shielding due to multiple micro-cracking around or within clusters near the main crack tip, and crack deflection due to the local mixed mode crack driving force contribute to the toughening in a fine particle reinforced composite. However, in a coarse particle reinforced composite, the increased deformation energy consumption in the matrix between large particles or the increased fracture surface caused by particle cracking plays the predominant role in enhancing toughness when void formation in the matrix around individual particles is prevalent or when particle cracking is the controlling fracture mechanism. It might be due to the complicated toughening mechanisms that the model proposed by Rice and Johnson (i.e. eq. (4)), which correlates the fracture toughness to CTOD only, may not be applicable to predicting the fracture toughness in fine particle reinforced composites.

4.2 Crack initiation point

The crack initiation toughness was calculated in some papers by using the maximum load on the load-displacement curve assuming that the crack was nucleated at the maximum
In other papers, however, the crack initiation toughness was assessed by supposing that the crack was initiated at the abrupt change point of compliance change rate. Based on the present observation, it is noted that at the ACP, the main crack propagated for $\sim 3.5 \mu m$ in the coarse particle reinforced composite (Fig. 2(d)) and in the fine particle reinforced composite, the micro-cracks were formed in front of the main crack tip and connected with the main crack (Fig. 4(f)). However, at the maximum loading point, the main crack extended for $\sim 12 \mu m$ in the coarse particle reinforced composite (Fig. 2(e)) while the crack propagated for $40 \mu m$ in the fine particle reinforced composite (Fig. 4(h)). From this observation, it can be inferred that the ACP reflects the crack initiation point more truly than the maximum loading point.

5. Conclusions

Based upon the above in-situ SEM observations of fracture processes in the two 6061Al matrix composites respectively reinforced with fine SiC particles and coarse SiC particles, conclusions are drawn as follows:

(1) The crack initiation and propagation of the coarse particle reinforced composite were mainly associated with the formation of voids in the matrix around SiC particles ahead of the main crack tip, mutual coalescence of voids and their connection with the main crack. However, boundaries between SiC particle clusters and their surrounding matrix, and the matrix within clusters were responsible for the crack initiation and failure of the fine particle reinforced composite.

(2) The crack initiation points evaluated by compliance change rate method reflected the true crack initiation points in the two composites.

(3) The toughening mechanisms of particle clusters and large particles in a composite are different. The enhanced fracture toughness in the composite reinforced with clustered fine particles was attributed to crack branching formed between SiC clusters and their surrounding matrix, shielding and anti-shielding due to multiple micro-cracking near the main crack tip and crack deflection due to the local mixed mode crack driving force.

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