**Abstract**

The current research period has emphasized 6061 and 7090 aluminum alloys as composite matrices, both with particulate SiC reinforcements. As in our previous work, we have found the fracture behavior to be sensitive to both process variables (in this case, different consolidation methods by the two material producers) and to metallurgical variables (in this case, degree of aging). In particular, overaging these materials leads to precipitation at the interface between the matrix and the SiC, which in turn appears to encourage initiation and growth of microvoids at those interfaces, leading to enhanced ductile fracture.
FUNDAMENTALS OF INTERFACIAL STRENGTH IN COMPOSITE MATERIALS

ANNUAL REPORT
1 JULY 1986 - 30 SEPTEMBER 1987

Air Force Office of Scientific Research

Grant AFOSR-86-0238
(CMU Account 1-52033)

A.W. Thompson and I.M. Bernstein
Principal Investigators

Dept. of Metallurgical Engineering and Materials Science
Carnegie Mellon University
Pittsburgh, PA 15213

Report No. MEMS-ALC-11
16 November 1987
Fundamentals of Interfacial Strength in Composite Materials

First Annual Report

This is the first annual report on the subject program. The experimental materials are aluminum-alloy matrices with SiC reinforcements, at present as particulate reinforcements. The overall goal is to understand the behavior of such materials in terms of both processing variables and metallurgical variables. One of the tools being used for this purpose is the introduction of hydrogen into the material, which typically alters ductility and some aspects of fracture behavior, while being present in sufficiently small quantities that it cannot be regarded as an alloying element.

ABSTRACT

The current research period has emphasized 6061 and 7090 aluminum alloys as composite matrices, both with particulate SiC reinforcements. As in our previous work, we have found the fracture behavior to be sensitive to both process variables (in this case, different consolidation methods by the two material producers) and to metallurgical variables (in this case, degree of aging). In particular, overaging these materials leads to precipitation at the interface between the matrix and the SiC, which in turn appears to encourage initiation and growth of microvoids at those interfaces, leading to enhanced ductile fracture.

SUMMARY OF FUTURE WORK

Additional work on details of tensile fracture processes is needed to understand and quantify behavior, particularly as regards the role of the reinforcement particles as both initiation sites and fracture path participants. Additional transmission electron microscopy is also needed to reveal details of the overaging structures, together with underaged and peak-aged structures. At Carnegie Mellon, these studies will continue in the next period.

Crack growth behavior is also a topic area of interest, as the tensile fracture behavior becomes better understood. Toughness behavior, both with and without hydrogen, will be of particular interest. With one of the Principal Investigators (IMB) moving to the Illinois Institute of Technology, this portion of the work will be conducted there. Also, sustained load cracking in the presence of hydrogen, as well as cyclic loading with hydrogen and the behavior of short cracks, would be desirable investigations to extend the tensile studies. They will be carried out at Carnegie Mellon.
MATERIAL

Two Al-SiC\textsubscript{p} composites are being investigated in this study. The Al alloy matrices of the composites are 6061 (Al-Mg-Si) and 7090 (Al-Zn-Mg-Cu). Both composites are reinforced with 20 v/o particulate \(\alpha\)-SiC (SiC\textsubscript{p}). The "\(\alpha\)" designation indicates that the crystal structure of the reinforcement is mostly the hexagonal \(\alpha\) phase. The average particle size in the 6061-SiC\textsubscript{p} composite is 3-4 \(\mu\)m. In 7090-SiC\textsubscript{p}, there is a bimodal SiC size distribution, with a large peak at 3 \(\mu\)m and a smaller, broad peak at 9-11 \(\mu\)m. DWA Composite Specialties, Inc. manufactures both composites by the P/M process illustrated in Figure 1.

Representative microstructures of the as-received composites are shown in Figure 2. These photographs are from the T-S plane, perpendicular to the extrusion direction. Figure 3 presents the area fraction of reinforcement for each composite on three planes through a metallographic specimen. There appears to be some particle alignment with the extrusion direction in 6061-SiC\textsubscript{p}. It should be noted that the particles are not spherical, but the aspect ratio is usually less than three.

The heat treatments used in this study are listed in Table 1. Temperatures chosen are similar to those used commercially; aging curves for each composite provided the aging times for under-aged, peak-aged and over-aged conditions.

RESULTS AND DISCUSSION

The composite materials have been tensile tested in each condition with the tensile axis parallel to the extrusion direction. Tensile
properties are presented in Table 2 where they are compared with properties of the monolithic matrix alloys. The presence of SiCp reinforcement increased the strength levels in both composites over the monolithic material and greatly decreased the ductility. It has been found that the proportional strength increase is less in composites with a stronger matrix. This was evidenced in 6061-SiCp and 7090-SiCp, where 7090 is the stronger monolithic alloy.

Fracture initiation in both composites occurred most often (eight of eleven tensiles) at large intermetallic particles. Figure 4 shows a fracture initiation site in 7090-SiCp. The intermetallics ranged in size from 45-150 μm. These intermetallics are the result of DWA's processing technique. Consolidation of DWA's composites is conducted at supersolidus temperatures; local melting occurs in the matrix, which allows intermetallic phases to form. Alcoa also makes P/M composites of this general type, but their processing method utilizes consolidation at sub-solidus temperatures. Tensile fracture in Alcoa specimens usually initiates at clusters of SiC particles or a very large reinforcement particle—rarely at intermetallics.

Examples of the tensile fracture surfaces of 6061-SiCp and 7090-SiCp are shown in Figure 5. Cracked SiC particles are evident on the fracture surfaces. Figure 6 shows matching fracture surface halves with matching halves of some particles indicated on the photograph.

Quantitative analysis of the fracture surfaces was performed to examine the area fraction of cracked SiCp on the fracture surface. Figures 7 and 8 show the results obtained for the various conditions of the composites. In both 6061-SiCp and 7090-SiCp, the area fraction of cracked SiC particles on the tensile fracture surfaces decreased as the material was aged. Work by Lewandowski, Liu, and Hunt(1) showed that in a matrix alloy very similar compositionally to 7090, a brittle precipitate forms at the SiC particle/matrix interface in the over-aged condition (Figure 9). We believe that this is also occurring in 6061-SiCp and 7090-SiCp. In the under-aged and peak-aged condition, a crack traveling through the material would find it easier to

propagate through particles in its path rather than around them. When precipitation occurs at the interface, propagation through the brittle precipitate may be easier than through the SiC particle. This explains why the area fraction of cracked SiC on the fracture surfaces decreases in the over-aged condition; more decohesion of particles from the matrix is occurring. Figures 10 and 11 show instances of decohered particles in the over-aged composites.

The tensile fracture surfaces of both composites were quantitatively examined to determine the frequency distribution of particle sizes for the cracked SiC particles on the fracture surfaces. For 6061-SiC_p (Figure 12), the cracked particle size distribution on the fracture surfaces was similar to the size distribution found on a T-S plane through a metallographic specimen for both under-aged and over-aged conditions. The situation is somewhat different for 7090-SiC_p (Figure 13). First, the proportion of large SiC particles found on the tensile fracture surfaces is higher than the proportion of large particles found on a T-S plane through the as-received material. (Recall that 7090-SiC_p has a bimodal particle size distribution.) Lewandowski, Liu, and Hunt(2) have proposed that this may be due to the greater probability of finding critically sized defects in the larger particles. Second, the proportion of cracked smaller particles on the fracture surfaces decreases as the 7090-SiC_p composite is aged. This may indicate that the matrix begins to play a more dominant role in the fracture process as precipitation occurs in the matrix during overaging. More tests will be necessary to confirm this.

CONCLUSIONS

Fracture initiation in these Al-SiC_p composites is process-sensitive. By comparing DWA and Alcoa composites, it is evident that different methods of consolidation have resulted in a tendency for different types of fracture initiation sites.

Precipitation occurs at the SiC particle/matrix interface in the over-aged condition for 7090-SiC\textsubscript{p}, and most likely for 6061-SiC\textsubscript{p}. This leads to more particle decohesion from the matrix during failure. In the over-aged condition, the area fraction of cracked SiC particles found on the tensile fracture surface was lower than in either of the other two conditions.

The matrix appears to play a more dominant role in failure of 7090-SiC\textsubscript{p} than of 6061-SiC\textsubscript{p}. Size proportions of cracked SiC particles in over-aged and under-aged conditions of 6061-SiC\textsubscript{p} fracture surfaces were similar to those proportions found in the as-received composite. In 7090-SiC\textsubscript{p}, the proportions changed with heat treatment.

**FUTURE WORK**

Fracture profiles of the tensile specimens of both composites will be examined to accurately determine the ratio of cracked to decohered particles on the fracture surface. This ratio is expected to decrease as the composites are over-aged. In order to test the theory of critical defects on the fracture of SiC particles, an analysis using Weibull statistics will be completed. This will indicate whether the critical defect theory is an appropriate one in this case. The region of the SiC particle/matrix interface will be examined using a TEM to verify the presence of precipitates in this area in the over-aged composites. The precipitate structure in the matrix itself will also be examined. It would also be useful to look at the composites in a state just before failure; perhaps voids at the SiC particles or grain boundaries would be in evidence. Double-notch tensile tests will make this possible. By putting two notches in a tensile gage and tensile testing in the usual manner, failure will occur at one notch and material close to failure will be found at the other notch. Straining electrode tests will be conducted to look at the effects of hydrogen on the role of the SiC\textsubscript{p} reinforcement in fracture.
DWA Al-SiCp Processing Steps

1. Blend Al and SiCp powders
2. Cold compact
3. Degas
4. Consolidate at supersolidus temperature
5. Extrude

Figure 1.
Figure 2. Composite T-S metallographic plane
Figure 3. Area fraction SiCp in as-received composites.
Table 1

Heat Treatment

<table>
<thead>
<tr>
<th></th>
<th>6061-SiCp</th>
<th>7090-SiCp</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solution</td>
<td>530°C/1h</td>
<td>488°C/1h</td>
</tr>
<tr>
<td>Age u/a</td>
<td>175°C/0.5h</td>
<td>121°C/4h</td>
</tr>
<tr>
<td></td>
<td>/2h</td>
<td>/22h</td>
</tr>
<tr>
<td></td>
<td>/20h</td>
<td>/72h</td>
</tr>
</tbody>
</table>
Table 2: Tensile test results of composite materials compared to monolithic matrix alloys

<table>
<thead>
<tr>
<th>Material</th>
<th>0.2% (MPa)</th>
<th>UTS (MPa)</th>
<th>%elp</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061-SiC</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>u/a</td>
<td>395</td>
<td>487</td>
<td>5.6</td>
</tr>
<tr>
<td>p/a</td>
<td>427</td>
<td>500</td>
<td>4.3</td>
</tr>
<tr>
<td>o/a</td>
<td>405</td>
<td>461</td>
<td>3.1</td>
</tr>
<tr>
<td>6061-0%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>p/a</td>
<td>276</td>
<td>311</td>
<td>12</td>
</tr>
<tr>
<td>7090-SiC</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>u/a</td>
<td>684</td>
<td>767</td>
<td>1.9</td>
</tr>
<tr>
<td>p/a</td>
<td>723</td>
<td>784</td>
<td>1.3</td>
</tr>
<tr>
<td>o/a</td>
<td>722</td>
<td>762</td>
<td>0.6</td>
</tr>
<tr>
<td>7090-0%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>p/a</td>
<td>586</td>
<td>627</td>
<td>10</td>
</tr>
</tbody>
</table>
Particle in 7090-SIC
Fracture initiation site at intermetallic

Figure 4.
Figure 5. Composite tensile fracture surfaces
Figure 6. Matching tensile fracture halves of over-aged 7090-SiC; matching halves of cracked particles indicated by arrows.
Figure 7. Cracked SiC on 6061-SiC fracture surfaces
Figure 8. Cracked SiC on 7090-SiC fracture surfaces
Figure 9. TEM micrograph showing $\eta$(MgZn$_2$) precipitates at the SiC particulate/MB78 matrix boundary in the T7X3 (over-aged) condition (from Lewandowski, Liu and Hunt, Microstructural Effects on the Fracture Micromechanisms in 7xxx Al P/M-SiC Particulate Metal Matrix Composites, Alcoa Laboratories Technical Report, June 19, 1987)
Figure 10. Matching tensile fracture halves of over-aged 6061-SiC; decohered particle and its cavity indicated by arrows
Figure 11. Matching tensile fracture halves of overaged 7090-SiC; decohered particle and its cavity indicated by arrows.
Figure 12. Frequency distribution of cracked particle sizes on fracture surfaces of 6061-SiCp
Figure 13. Frequency distribution of cracked particle sizes on fracture surfaces of 7090-SiCp.