INTERFACE MODIFICATION OF POLYMER-DERIVED SiC SINGLE-FIBER AND Ti ALUMINIDE COMPOSITES

T. Suzuki, H. Umehara, and X.L. Guo

National Institute of Materials and Chemical Research
1-1 Higashi, Tsukuba, Ibaraki 305-8565, Japan

SUMMARY: SiC-based-fiber-reinforced Ti aluminide (TiAl) composites whose interfaces were modified by C, BN, W, and Mo coatings were prepared by thermal decomposition of a hydrocarbon, of BCl₃ and NH₃, and by sputtering. The interface structure and composition were analyzed by scanning electron microscopy, electron probe microanalysis and transmission electron microscopy. The tensile strength properties of composites were tested using model TCM-50 and determined by Weibull analysis. Results showed that the interface between the SiC-based fiber and TiAl matrix was modified markedly by C, BN, W, and Mo coatings compared to non-coated SiC-based-fiber-reinforced TiAl composites. C enrichment by coatings strongly affected interface modification. Of the modified coatings used, W improved the stability of SiC-based-fiber-reinforced TiAl composite interfaces the best, especially above 1200 K.

KEYWORDS: polymer-derived SiC-based fiber, Ti aluminide, tensile strength, interface, C, BN, Mo, W coating, compatibility.

INTRODUCTION

Ti aluminides feature high specific strength and heat resistance (1300 K), making them potentially useful at 1000-1400 K in aeronautics and space applications [1]. Their drawbacks of inadequate ductility and toughness are overcome by fiber reinforcement. Since the intermetallic compound TiAl as Ti is chemically active, TiAl composites are usually formed at comparatively low temperatures. Because the processing temperature is generally >1300 K, reactions must be avoided at fiber/matrix interfaces, meaning that interfaces must be modified. TiAl have been synthesized on heat-resistive fibers with low oxygen content and appropriate adhesion by controlled sputtering [2-4]. We studied optimum coating to improve SiC/TiAl composite interface properties and discuss on the mechanisms involved in improving composite tensile strength. Cracks generated by coefficient of thermal expansion (CTE) mismatch is a significant problem in composites, especially reinforced with extra-large diameter (120 x 10⁻⁶ m) fibers. The problem, however, will be solved by using small-diameter fibers such as polymer-derived SiC.
EXPERIMENTS

We studied two types of polymer-derived SiC fiber, SiC and SiTiC (Table 1). The surfaces of as-derived fibers were modified with C, BN, W, and Mo coatings, selected to minimize the mismatch of the thermal expansion coefficient, high-temperature interface reactivity and optimize interfacial bonding strength between the fiber and matrix (Table 2).

Table 1: SiC-based fibers

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Diameter ($10^{-6}$m)</th>
<th>Oxygen content (Ti content) (mass%)</th>
<th>Fabrication</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si-C-O fiber (SiC)</td>
<td>14</td>
<td>0.5 (0)</td>
<td>Polycarbosilane pyrolyzation</td>
</tr>
<tr>
<td>Si-Ti-C-O fiber (SiTiC)</td>
<td>11</td>
<td>5 (2)</td>
<td>Polytitanocarbosilane pyrolyzation</td>
</tr>
</tbody>
</table>

Table 2: Modification coatings of SiC and SiTiC fibers

<table>
<thead>
<tr>
<th>Coating</th>
<th>Thickness ($10^{-6}$m)</th>
<th>Fabrication</th>
<th>Temperature (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.1</td>
<td>Hydrocarbon thermal decomposition</td>
<td>1000</td>
</tr>
<tr>
<td>BN</td>
<td>0.1</td>
<td>BCl$_3$ and NH$_3$ decomposition</td>
<td>1100</td>
</tr>
<tr>
<td>W</td>
<td>0.5</td>
<td>Sputtering</td>
<td>300</td>
</tr>
<tr>
<td>Mo</td>
<td>0.5</td>
<td>Sputtering</td>
<td>300</td>
</tr>
</tbody>
</table>

TiAl 2 x $10^{-6}$ m thick was deposited on as-derived and modified fibers by RF magnetron sputtering at an output of 200 W [2-4]. The coating and fracture morphology of composites were observed using scanning electron microscopy (SEM) and transmission electron spectroscopy (TEM)(300 kV). The tensile strength properties of the single-fiber-reinforced TiAl composites were measured by using a tensile machine model TCM-50 (NMB Co.). The tensile speed was 5 x $10^{-4}$ m/min. More than 20 specimens were applied for each. Weibull analysis was used to determine tensile strength parameters such as mean and variation coefficients of single-fiber-reinforced composites [2-6]. The interface composition of composites was determined using electron probe microanalysis (EPMA) and energy-dispersive x-ray analysis (EDX)(probe: 1nm).

RESULTS AND DISCUSSION

Cross-sections of single-fiber-reinforced TiAl composites fixed by epoxy resin were polished or fractured and observed using SEM (Fig. 1), and showed a homogeneous interface between the SiC fiber and TiAl matrix formed around SiC fiber coated with W (Fig. 1(a)) and Mo (Fig. 1(b)). The TiAl matrix also formed homogeneously around SiC fiber.
Fig. 1: Cross-sectional morphologies of single-fiber-reinforced TiAl composites: (a) SiC/W/TiAl and (b) SiC/Mo/TiAl.

Fig. 2: Tensile strength of single-SiC-fiber-reinforced TiAl composites, (above) and SiTiC-reinforced TiAl composites, (below). m: modifier.
The tensile strength of SiC- and SiTiC-fiber-reinforced TiAl composites changed with increasing temperature (Fig. 2). Compared to modified fiber-reinforced TiAl composites, the tensile strength of as-derived fiber-reinforced TiAl composites was lower and decreased with increasing temperature, presumably because of the resistance of C, BN, Mo, and W coatings to the interface into fibers [2, 3, 7]. It was found, however, that composite tensile strength is influenced by sputtering power [2, 3]. C and BN coatings markedly improved the strength of fiber-reinforced TiAl composites up to 1100-1200 K. This was increasingly conspicuous with increased sputtering power. C and BN coatings act as weak-interface-modifiers for microcrack propagation and barrier layers, inhibiting reactions at the fiber and TiAl interface. The tensile strength of BN-coated SiC fiber-reinforced TiAl composites was higher than that of C-coated SiC fiber reinforced TiAl composites after heat treatment at 1073 K for 2 hr in Ar. The tensile strength of SiC-based-fiber-reinforced TiAl composites modified by refractory metal coatings of W and Mo did not improve markedly up to 1100 K, but the deterioration was inhibited above 1100 K. Fibers extracted with chemicals after heating showed less deterioration at a low oxygen content, but significant deterioration at a high oxygen content, indicating that oxygen in fibers affected interface reactions. We found that the markedly higher strength of C-coated SiC-based-fiber-reinforced TiAl in SiC fiber up to 1100 K, and in SiTiC fiber up to 1200 K than that of W/C or W/BN double-coated SiC-based-fiber-reinforced TiAl. The heat resistivity of SiTiC fiber containing a few percent of Ti was almost the same as the SiC fiber having low oxygen content (0.5%). Cross-sectional observation of BN-SiC fiber-reinforced and C-SiTiC fiber-reinforced TiAl composites after heating (Fig. 3) showed that fiber/TiAl interfaces were comparatively well defined and had no obvious difference from before heating except that the C intensity decreased slightly (Fig. 4).

TEM observation [8, 9] to determine the advantages of C-coated-SiTiC reinforced TiAl composites in detail (Fig. 5 and Fig. 6) showed a Ti-enriched layer at the interface heated at 1200 K in Ar, indicating that controlling the reaction of Ti and coating materials as a modifier at the interface is the key to improving the mechanical properties of composites.

![Image](a) BN-SiC/TiAl and (b) C-SiTiC/TiAl before heat treatment.

Fig. 3: Cross section and fracture morphologies of modified-fiber-reinforced composites (a) BN-SiC/TiAl and (b) C-SiTiC/TiAl before heat treatment.
CONCLUSION

C, BN, Mo, and W coatings are compatible with the SiC surface and homogeneously coated the fiber surface. Interface reactions between the SiC fiber and TiAl coating, are inhibited by homogeneous C, BN, Mo, and W coatings. C enrichment in the interface greatly affects interface modification up to 1100-1200 K. Of the modified coatings we studied, W improves the stability of SiC-based-fiber reinforced TiAl composites best at the temperature above 1200 K.
Fig. 6: EDX analysis at points (a) 1, (b) 2, (c) 3, and (d) 4 in Fig. 5.

REFERENCES


