Preliminary Characterization of Ion-Plated Aluminum/Graphite Composites

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### PRELIMINARY CHARACTERIZATION OF ION-PLATED ALUMINUM/GRAFITE COMPOSITES

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Ion-plated aluminum/graphite (Al/Gr) composite material has been characterized in terms of microstructure, tensile properties, and fractography. Longitudinal and transverse tensile strengths were found to be superior to those of Al/Gr composites fabricated by liquid metal infiltration. More importantly, the Al/Gr precursor tape was thin and flexible enough to allow cross-ply reinforcement and selective reinforcement in complex shapes.
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INTRODUCTION

Two metal matrix composites having considerable tri-service importance are graphite fiber reinforced aluminum (Al/Gr) and magnesium (Mg/Gr). Current and projected applications range from portable assault bridges through aircraft, missile, and space applications. Manufacturing Al/Gr composite plate in this country is a complex, somewhat labor-intensive process that begins with coating the graphite fiber yarn (tow) with titanium diboride, for example, by chemical vapor deposition (CVD) to promote wetting by molten aluminum. The coated fiber bundles are passed through a liquid metal bath to impregnate the yarn which, when cooled, becomes an Al/Gr wire that is typically 1-1.5 mm in diameter. The thickness and stiffness of these wires make conventional filament winding and mat layup techniques impractical, if not impossible. The usual next step is to encapsulate the Al/Gr wires in aluminum foil and arrange them in parallel arrays suitable for hot-press diffusion bonding to produce the final plates of Al/Gr composite material.

The limitations imposed on structural applications of such composites solely by the final material configuration are substantial and provide a strong incentive to explore alternative fabrication techniques. Further impetus comes from the need to increase substantially both the fiber volume fraction and the transverse tensile strength of state-of-the-art Al/Gr composites. From a practical standpoint, the most versatile composite precursor material for continuous reinforcement would be in the form of sheet goods, that is, a thin and flexible continuous tape or ribbon of Al/Gr composite that would be amenable to cross-plying and to configuring into complex shapes.

Many techniques have been developed to join constituent materials into a metal matrix composite. Ion plating is one such technique that appears not to have been adequately exploited in spite of the knowledge that it produced graded interfaces that were usually fully dense and strongly adherent. The kinetics of the interface formation are not well understood, but four major factors in controlling the process have been suggested, namely, (a) sputter etching of the substrate, (b) high kinetic energy of the evaporant, (c) amount of ionized evaporant, and (d) surface heating effects that both enhance diffusion and facilitate surface reactions.1

Recently, an ion-plating technique was used in Japan to produce an Al/Gr ribbon of reasonable quality. The ribbons were simply stacked and hot-press diffusion bonded to form thin plates of composite material. This report evaluates in a preliminary and selective fashion the engineering properties and potentials of this material and process. The reported engineering mechanical properties of the ion-plated Al/Gr composite were at least as good as

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those of Al/Gr composites fabricated by the liquid metal infiltration process. However, no direct measure of transverse tensile strength was reported, and scant support for the claim of uniform reinforcement was found.

MATERIAL EVALUATION

A small quantity of the Japanese ion-plated Al/Gr composite was obtained recently in the form of a thin consolidated plate as well as a continuous precursor tape. The flexible tape was nominally 25 μm thick by 8 cm wide, easy to handle and shape, and apparently quite durable. Such tapes were stacked and consolidated by hot-press diffusion bonding to produce the composite plate material which was 10 cm square by 3.4 mm thick. Fabrication process parameters were not available. Moreover, it was not known to what extent these samples represented typical Al/Gr composite material produced by the Japanese ion-plating process. The available material was just sufficient to perform a preliminary characterization of microstructure, tensile properties, and fractography, the results of which are described separately below.

Microstructure

Metallographic specimens of the ion-plated Al/Gr precursor tape and the diffusion-bonded composite plate were prepared by conventional techniques. The microstructure of the tape is illustrated in Figure 1, which shows a segmented partial cross-section (r ~ 2%). There was substantial variability in tape thickness, which ranged from about 3 μm to as much as 40 μm. The irregular fiber spacing could pose problems in consolidating a uniform composite plate.

The microstructure of the consolidated Al/Gr plate is shown in Figure 2. Matrix composition was analyzed by direct current argon plasma atomic emission photometry (DCP) and found to consist of aluminum plus the following percentages of other elements:

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>0.15</td>
</tr>
<tr>
<td>Hg</td>
<td>0.02</td>
</tr>
<tr>
<td>Cu</td>
<td>0.03</td>
</tr>
<tr>
<td>Mg</td>
<td>0.22</td>
</tr>
<tr>
<td>Mn</td>
<td>0.12</td>
</tr>
<tr>
<td>Pb</td>
<td>0.04</td>
</tr>
<tr>
<td>Ba</td>
<td>0.36</td>
</tr>
<tr>
<td>Cr</td>
<td>0.04</td>
</tr>
</tbody>
</table>

The analysis also included B, P, Cd, Ca, Co, Zn, Ni, and Ti. These elements, if present, were below the detection limits of the equipment (i.e., < ppb). This matrix was unidirectionally reinforced with approximately 38 volume percent of high tenacity (HT) graphite fiber that averaged 7 μm in diameter. A moderately good dispersion of graphite fibers was obtained, but Figure 2A shows an obvious tendency toward undesirable clustering of fibers. Closer examination of typical clusters revealed large numbers of unplated fibers and nearly continuous low-energy fracture paths through the composite, as shown in Figure 2B. These unconsolidated regions extended axially in the composite for considerable distances as shown in Figure 2C. However, the consolidation and interface morphology were quite good away from the clusters, as may be seen in Figures 2D and parts of 2B. The clustering of fibers was most likely due to poor control of thickness uniformity in the Al/Gr precursor tape shown in Figure 1.
FIGURE 2. Optical Metallographs of Polished Specimens of Al/Sr Composite. Cross-Sections Oriented Perpendicular (A, B) and Parallel (C, D) to the Fiber Reinforcement Direction.
Tensile Properties

Mechanical properties of the Al/Gr precursor tape were not evaluated. The composite plate stock was ground into conventional flat tensile coupons having reduced-width gage sections that were 36 mm long, 9 mm wide, and 3.4 mm thick. Three longitudinal and three transverse specimens were prepared. Each was fitted with conformal shoulder tabs of 0.8 mm-thick aluminum, which were bonded to the specimen with epoxy, to facilitate gripping the specimen in the tensile testing machine. Strain gages were mounted on both sides of the gage section to eliminate effects of specimen bending in the strain indications. The specimens were then loaded to fracture at quasistatic strain rates ($\varepsilon = 10^{-4} \text{s}^{-1}$).

The results of these room temperature tensile tests are presented in Table I. Corresponding data are provided for Al/Gr composite material made by the hot-press diffusion bonding of Al/Gr wires that were fabricated by the liquid-metal-infiltration technique. The ion-plated Al/Gr composite utilized a lower modulus graphite fiber, resulting in a slightly smaller Young's modulus for the composite. The transverse Young's moduli were virtually identical. The tensile strength of the ion-plated material was slightly greater than that reported for liquid-metal-infiltrated material in the longitudinal direction and significantly greater in the transverse direction.

Table I. TENSILE PROPERTIES OF ALUMINUM/GRAPHITE COMPOSITE PLATES

<table>
<thead>
<tr>
<th>Composite Precursor Material</th>
<th>Longitudinal</th>
<th>Transverse</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Young's Modulus GPa (MSI)</td>
<td>Ultimate Tensile Strength MPa (KSI)</td>
</tr>
<tr>
<td>Ion-Plated Al/Gr Tape</td>
<td>97-117 (14-16)</td>
<td>669 (97)</td>
</tr>
<tr>
<td>Liquid-Metal-Infiltrated Al/Gr Wire</td>
<td>110-131 (16-19)</td>
<td>565 (82)</td>
</tr>
</tbody>
</table>

NOTE: Precursor materials were consolidated into unidirectionally reinforced plates having 35-40 volume percent fibers by conventional hot-press diffusion bonding.
Fractography

The failure surfaces of these tensile specimens were examined morphologically in a scanning electron microscope, and typical fractographs are presented in Figure 3. In longitudinal tension, the composite failed by fiber fracture and pull-out as shown in Figure 3A. A detailed view of a pulled-out fiber in Figure 3B shows good adhesion of the matrix where plating was adequate and at least vestigial matrix where it was not. In transverse tension, Figures 3C and 3D show a fair amount of fiber/matrix interface decohesion. It was not clear whether this failure behavior was due to an absence of ion-plated aluminum on significant portions of the fibers in the tapes or whether it was due to poor fiber wetting (or fiber/matrix interface reaction).

SUMMARY AND CONCLUSIONS

A preliminary characterization of ion-plated aluminum/graphite composite material was completed in terms of metallography, tensile properties, and fractography. The precursor material was continuous ion-plated Al/Gr tape that was flexible and easily handled. Large fluctuations in tape thickness led to excessive clustering of fibers in the final composite plate which was fabricated simply by hot-press diffusion bonding these tapes together. Longitudinal and transverse tensile strengths and moduli were measured and compared quite favorably to those reported for liquid metal infiltrated Al/Gr composites. SEM tensile fractographs disclosed probable sources of weakness in the composite that could be attributed to poor control of Al/Gr precursor tape thickness and perhaps to poor fiber wetting or inadequate fiber/matrix interface reactions.

Several important observations were made:

(a) The Al/Gr precursor tape was thin enough to make cross plying practical, as well as flexible enough to consider complex shapes and selective reinforcement schemes.

(b) The high probability of attaining 50-60 volume percent fiber loading provides a realistic expectation of zero coefficient of thermal expansion (CTE) in structures for space applications.

(c) In spite of obvious microstructural deficiencies, the transverse tensile strength is already superior and potentially outstanding.

(d) The physics and mechanics of forming a graded interface by ion plating are poorly understood. The process has been applied to the fabrication of Al/Gr composites with limited success, but the enormous potential payoff for successful development of the technique warrants more extensive research and development.

In general, it was concluded that the ion-plating process for fabricating Al/Gr composites has great potential to provide high-performance composites in versatile configurations that would facilitate their use in critical military structural applications. There is nothing inherent in the process to limit it to Al/Gr, and this method may lead to the elusive magnesium/graphite composite in practical quantities and configurations, as well as to a variety of other tactically and economically important composite systems.
FIGURE 3. SEM Tensile Fractographs of Failure Surfaces in Al/Gr Composite. Tensile Loading Axis Parallel (A, B) and Perpendicular (C, D) to the Fiber Reinforcement Direction.
REFERENCES


3. Material provided by Dr. S. L. Channon, Institute for Defense Analyses, Arlington, VA

