Development of Intelligent Processing Methodology for Intermetallic Matrix Composites

Progress Report
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1.0 INTRODUCTION

The project aims at development of processing technologies for a broad, new family of in-situ metal matrix composites based upon the innovative use of multilithic reinforcement strategies. Intermetallic matrix composites (IMCs), reinforced with a dispersed ceramic phase, will be incorporated into metallic matrices to serve as reinforcing entities within the resulting multilithic reinforced composite (MRC). IMC-reinforcement in metallic matrices is particularly novel since they can be created to possess low temperature strengths normally unique to structural ceramics, and retain a metallic-like ability to be deformed at high temperatures. When combined with creative processing methodologies, such composites will offer an unprecedented degree of microstructural and property design capability. When specifically applied to light-metal matrices, the composites will possess the normally elusive combination of high specific strength, thermomechanical stability, economy of processing, and increased use-temperature capability.

While the concept of an IMC-reinforced metal matrix composite can be broadly extended to a wide range of conceivable processing methodologies and composite geometries, deformation processing techniques has been selected for this effort as the approach whereby the best properties of both the IMC and matrix components can be most efficiently and synergistically applied. For example, through the imposition of high temperature, powder-based extrusion, an aligned MRC can be created if the metal matrix and the IMC-reinforcement deform commensurably.

2.0 OBJECTIVES

The specific objectives of this study are to:

1. Identify most significant MRC material system(s) for advanced navy propulsion systems.

2. Develop and demonstrate feasibility of using advanced material process modeling to establish the conditions of extrusion whereby commensurate flow of a metal and a discontinuously-reinforced IMC can be achieved.

3. Scale-up process to determine the most cost-effective deformation processing route (extrusion, forging) for production of MRC materials.

4. Perform market analysis to establish a database for commercialization of MRC materials.
3.0 PROGRESS AND STATUS

Task 1. Identification of Most Significant MRC Materials Systems for Advanced Navy Propulsion Applications

As part of the MATSYS effort to identify MRC material systems of greatest interest, discussions have continued between MATSYS and Pratt & Whitney to define the MRC materials and properties. Based on these discussions, the matrices of strongest interest are Ti-6-2-4-2 as well as either a 2xxx or 6xxx aluminum matrix. Discussions concerning the selection of reinforcing phase(s) are continuing, with significant interest expressed in Al3Ti and rare earth variants (e.g., scandium) intermetallics reinforced with either TiB2, B4C or SiC. A major interest expressed by Pratt and Whitney would be the development of an aluminum material system with application capabilities in the temperature range of 500°C. Such MRC systems could potentially serve as a broad-based replacement for more expensive titanium alloys currently in use in inlet and first stage compressor sections of the gas turbine engine.

MATSYS and Pratt and Whitney will continue to explore the feasibility of these systems, with the goal of providing an initial feasibility demonstration in the next few months.

Task 2. Process Model Development, Implementation and Validation

To date the effort has focused upon co-deformation processing of metal/IMC blends based upon two variants of the Ti-6Al-4V + (Ti-Al + TiB2) MRC material system. The objective of this activity is to determine the processing conditions whereby an IMC will commensurately deform when processed within a ductile metal matrix. The primary objective of this task is the development of a computer model with the ability to predict the deformation behavior of the reinforcing phase in a metal matrix composite. To assess the validity of the model several extrusions were performed at Imperial College.

Extrusion trials. MRC constituent powders were prepared and blended at Virginia Tech, and hermetically sealed via electron-beam welding (at KTI, Inc. in East Windsor, CT) in preparation for extrusion-processing using the Processing Laboratories at Imperial College. Building upon the successful extrusions produced from the Phase I effort, these compositions likewise focused on Ti-6Al-4V matrices reinforced with discrete Al3Ti/TiB2 IMC particles. Nominal MRC compositions, their designations, and the extrusion conditions are shown in Table I. The specific formulations attempt to discern the roles of volume percentage of IMC in the MRC (T-2040, T-3040, and T-4040), volume fraction of TiB2 in the IMC (T-4000, T-4020, and T-4040), and ultimately, whether a
strong bond between the IMC and Ti-6Al-4V (established via subjecting the billet to hot isostatic pressing (HIP) prior to extrusion processing) affects co-deformability. Three independent variables were considered in the design of the extrusion experiments, namely, extrusion temperature, v% IMC and v% of TiB₂ in IMC.

Previously, extrusion temperatures above 1200°C revealed that the Al₃Ti intermetallic may be vulnerable to melting due to heating during extrusion. Thus, a component of this extrusion campaign also included an attempt to assess whether a lower temperature bound could be defined (e.g., 1180°C) to avoid the potential and detrimental melting of the IMC. However, model predictions of temperature profiles throughout the extrusion process revealed that initial billet temperatures of 1200°C would result in a steady-state temperature of approximately 1100°C in the deformation zone. This is well below the melting temperature of the Al₃Ti (~1350°C), and thus, should not be a contributing factor in the selection of the processing conditions. The melting which did occur during previous trials is believed to have occurred as a consequence of excessive adiabatic heating attributable to not using a die lubricant.

Extrusion trials were conducted to assess whether a high strength IMC is capable of commensurate deformation within a metallic matrix. Results of the co-deformation trials are illustrated in Figures 1 through 3. The trials demonstrated that a high strength, high aspect ratio IMC “fiber” can be produced in-situ within a metallic matrix. Other microstructural features observed in the extruded specimens include lack of reaction at matrix-IMC interface, stability of the IMC within the Ti-6Al-4V matrix, stability of the TiB₂ particles within the Al₃Ti and some residual porosity within the IMC particles.

Microstructural and mechanical characterization of the extruded billets is now ongoing. Characterization will include an assessment of the co-deformation (aspect ratio of the deformation-processed IMC), residual porosity measurements, and mechanical behavior (room and elevated tensile or compression testing).

Elimination of the porosity can be achieved by:

1. HIPping the powder billet first. This will establish a strong powder-powder bonding and may lead to more effective load transfer to the IMC, thus increasing its flow and densification.

2. Use of a higher strength matrix, such as a high strength Ti alloy like Ti-6-2-4-2. This will increase the flow stress in the matrix and hence the stress on the IMC.
3. Reduction of the IMC powder size to reduce “void volume” associated with the IMC sponge.

**Table I.**
Designation, nominal composition and extrusion parameters for each of the extrusion trials performed at Imperial College.

<table>
<thead>
<tr>
<th>Extrusion Designation</th>
<th>Composition</th>
<th>Initial Billet Temperature (°C)</th>
<th>Nominal Extrusion Die Ratio</th>
<th>Independent Variable</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-3040</td>
<td>Ti-6Al-4V + 30 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1180</td>
<td>12:1</td>
<td>Extrusion temperature</td>
</tr>
<tr>
<td>T-3040</td>
<td>Ti-6Al-4V + 30 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1200</td>
<td>12:1</td>
<td></td>
</tr>
<tr>
<td>T-4040</td>
<td>Ti-6Al-4V + 40 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1200</td>
<td>12:1</td>
<td>v% IMC; Ti-6Al-4V / IMC strain distribution</td>
</tr>
<tr>
<td>T-3040</td>
<td>Ti-6Al-4V + 30 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1200</td>
<td>12:1</td>
<td></td>
</tr>
<tr>
<td>T-2040</td>
<td>Ti-6Al-4V + 20 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1200</td>
<td>12:1</td>
<td></td>
</tr>
<tr>
<td>T-4040</td>
<td>Ti-6Al-4V + 40 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1200</td>
<td>12:1</td>
<td>v% TiB₂ in IMC; strain in Al₃Ti</td>
</tr>
<tr>
<td>T-4020</td>
<td>Ti-6Al-4V + 40 v% (Al₃Ti + 40 v% TiB₂)</td>
<td>1200</td>
<td>12:1</td>
<td></td>
</tr>
<tr>
<td>T-4000</td>
<td>Ti-6Al-4V + 40 v% Al₃Ti</td>
<td>1200</td>
<td>12:1</td>
<td></td>
</tr>
</tbody>
</table>

Based upon the results of those trials, it is now appropriate to examine the numerous means by which the composite microstructure can be optimized and customized for specific applications or performance needs. Specifically, analysis is required which will allow one to define processing and microstructural conditions whereby the homogeneity of deformation can be maximized across the cross section of all present components and particles. Further, the ability to specifically “design” the fibrous IMC introduces the ability to create composites with broad property (e.g., strength, modulus, ductility, toughness) ranges. For example, co-deformability will be influenced by the volume percentage, size, and flow stress of the IMC; the latter can be independently scaled by varying the matrix composition, the volume percent of TiB₂, and the temperature and rate of processing. The resulting MRC properties will rely upon the properties and distribution of the IMC incorporated.
Figure 1. Ti-6Al-4V + 40 v% (Al, Ti+40 v% TiB$_2$) MRC produced via extrusion at 1200°C and an extrusion ratio of 12:1. As shown, the IMC has deformed with the metallic matrix.

Figure 2. Microstructural features of MRC. TiB$_2$ is stable within the IMC reinforcement and no reaction layer is observed between the IMC and metallic matrix.
Model development. The development of the computer model began with the application of the deformation simulation software FORGE2. Dimensions of an extrusion die, container, and extrusion ram were taken from the mechanical drawings of such tools and transferred into the computer model. Several assumptions regarding the billet were made to simplify the model. First, the billet was modeled as a solid block of material, without the mild steel canister which contains the actual powder. The steel canister will be incorporated by way of the friction developed on the die walls during extrusion, as such the steel can was treated as an imperfect lubricant during the extrusion. It was further assumed that the mechanical and physical properties of the composite (matrix plus IMC reinforcement) could be adequately estimated by a simple rule-of-mixtures approach. This information is subsequently applied to the Norton-Hoff equation (Eq. 1) which describes the mechanical portion of the deformation process. Physical properties, i.e., density, specific heat, and thermal conductivity, are shown with the Norton-Hoff constants in Table II. The Norton-Hoff equation parameters (n, m, K, and b) were determined in the Phase I effort from the deformation behavior of the IMC in monolithic form and from Ti-6Al-4V data found in the literature.

\[
\sigma_{\text{flow}} = \sqrt{3}^{m+1} \cdot K \cdot \varepsilon^n \cdot \dot{\varepsilon}^m \cdot \exp \left[ \frac{\beta}{T} \right]
\]

Eq. 1
Two extrusion simulations were produced for the Ti-6Al-4V + 40 v% (Al₃Ti + 40 v% TiB₂) MRC. Both simulations incorporated an initial billet temperature of 1200°C, a tool temperature of 500°C, and an extrusion ram speed of 2.7 mm/sec. All these conditions are consistent with the experimental conditions actually imposed in extrusion trials. The thermal properties of the constituents were used to account for cooling of the extruded rod as it was exposed to ambient temperatures. The difference between the two simulations lay in the assumption of the frictional behavior, i.e., the amount of friction between the walls of the extrusion die and the billet. The first case assumed that there was no friction at these interfaces, while the second model assumed complete sticking friction.

Table II.
Properties of Ti-6Al-4V and the Al₃Ti + 40 v% TiB₂ IMC. Also shown are estimated values (rule-of-mixture) for a composite comprised of 40 v% of the IMC within a matrix of Ti-6Al-4V (T-4040).

<table>
<thead>
<tr>
<th>Property</th>
<th>Ti-6Al-4V</th>
<th>Al₃Ti+40 v% TiB₂</th>
<th>T-4040</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strain sensitivity, n</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Activation energy, b (K⁻¹)</td>
<td>9,530</td>
<td>13,160</td>
<td>0</td>
</tr>
<tr>
<td>Constant, K</td>
<td>0.023276</td>
<td>0.004738</td>
<td>0.015861</td>
</tr>
<tr>
<td>Strain-rate sensitivity, m</td>
<td>0.148</td>
<td>0.208</td>
<td>0.172</td>
</tr>
<tr>
<td>Density, ρ (kg/m³)</td>
<td>4,428</td>
<td>3,800</td>
<td>4,178</td>
</tr>
<tr>
<td>Specific heat, Cₚ (kJ/mol)</td>
<td>959</td>
<td>N/A</td>
<td>959</td>
</tr>
<tr>
<td>Effusivity, b</td>
<td>8,243</td>
<td>N/A</td>
<td>8,243</td>
</tr>
</tbody>
</table>

The models will be assessed by using a feature of the computer software called a Materials Point History (MPH). The MPH contains all the stresses, strains, temperatures, and positions for a user-specified point as a function of time. Given this data it will be possible to track the stress, strain, and temperature as a function of the strain-rate and compare this to a similar position in the actual extrude. A comparison of the data from the computer model to the results of quantitative microscopy will then be used to appraise and improve the computer model.

Further modeling improvements will be implemented. Efforts are underway to establish a physically faithful process model to simulate extrusion. During the extrusion of an aggregate of powders it is necessary to consider the strain distribution within multiple phases. The modeling effort will identify conditions whereby the deformation of the two constituents will occur at similar rates within the extrusion process zone, thus leading to fully-compatible (fully-commensurate) co-deformation. Micromechanics, mechanism-based models will be used to simulate the extrusion process and optimize processing conditions. These models were developed for monolithic material and are being modified to enable simulation of composite materials. This effort will be coordinated with work
currently underway by Prof. Ashby and his colleagues where models are being developed for compaction of composite materials.

4.0 PLANNED ACTIVITIES

Discussions will continue with Pratt and Whitney to define the MRC materials and properties required to meet navy system requirements, and to specify components which would serve as candidates for MRC materials insertion. In addition, model development will continue and will be supported by characterization of MRC constituent materials.