DEFORMATION PROCESSED IMC-REINFORCED METAL MATRIX COMPOSITES

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(ABSTRACT)

The feasibility of utilizing TiB$_2$-reinforced near-gamma TiAl intermetallic matrix composites (IMCs) as a reinforcing entity within a commercially pure Ti matrix was investigated. IMCs are "ceramic-like" at ambient to moderate temperatures, and "metallic-like" in their deformation behavior above their brittle-to-ductile transition temperature, thus IMCs create opportunities to create unique in-situ composite microstructures otherwise unattainable using conventional ceramic reinforcements.

CP titanium composites reinforced with 20 vol% of near-gamma TiAl IMC were produced by powder blending and densifying via high temperature extrusion deformation processing. The microstructures of the in-situ processed composites were characterized in terms of size, aspect ratio and average spacing of the IMC reinforcement. The microstructural features were correlated to observed mechanical behavior of the composites relative to the unreinforced matrix. The results indicate that the strengthening is derived from microstructural changes within the matrix due to the presence of the IMC particles, and solid solution strengthening due to the diffusion of Al from the reinforcing IMC phase into the Ti matrix. The increase in flow strength due to the former contribution correlates with the inverse square root of the IMC interparticle spacing.
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O. OBJECTIVE AND SUMMARY OF RESEARCH

An unconventional approach to the development of metal matrix composites will be examined by exploring the processability and microstructural design of intermetallic-matrix composite (IMC)-reinforced titanium composites. That is, the feasibility of utilizing TiB$_2$-containing near-$\gamma$ titanium aluminide as a reinforcing entity within a titanium matrix will be investigated.

The mechanical properties of TiB$_2$-reinforced gamma titanium aluminide composites compare favorably with those of silicon carbide and alumina currently under consideration as reinforcement in metallic matrices. Table 1 illustrates that with sufficient volume fractions of discontinuous TiB$_2$ both the strength and modulus of the titanium aluminide composites approach the values exhibited by these more "conventional" reinforcement materials.

Thus, based upon these ceramic-like ambient temperature properties, it is anticipated that intermetallic matrix composites (IMCs) can be used as reinforcements in metal matrices to yield properties currently sought through the use of ceramic reinforcements.

While ceramic-like at ambient and moderate temperatures, IMCs possess the unique characteristic of becoming abruptly "metallic-like" in their deformation behavior above their usually well-defined brittle-to-ductile transition temperature. It is suggested that the ambient temperature ceramic-like behavior of IMCs, combined with their ability to undergo substantial deformation at high temperatures, presents opportunities to create unique in-situ composite microstructures not otherwise attainable using conventional ceramic reinforcements.
Table 0: Example mechanical properties of TiB₂-reinforced near-gamma titanium aluminides as compared to candidate structural ceramic reinforcements [1].

<table>
<thead>
<tr>
<th>Material</th>
<th>(\sigma_{\text{fracture, compression}}) (MPa)</th>
<th>Elastic Modulus (GPa)</th>
<th>(\sigma_{\text{fracture, tension}}) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiAl + 30 vol% TiB₂</td>
<td>1800(^3)</td>
<td>278(^3)</td>
<td>725(^3)</td>
</tr>
<tr>
<td>TiAl + 40 vol% TiB₂(^1)</td>
<td>2344(^2)</td>
<td>290(^2)</td>
<td>700(^2)</td>
</tr>
<tr>
<td>TiAl + 50 vol% TiB₂(^1)</td>
<td>2620(^2)</td>
<td>305(^2)</td>
<td>400(^2)</td>
</tr>
<tr>
<td>TiAl + 60 vol% TiB₂</td>
<td>2900(^2)</td>
<td>330(^2)</td>
<td>250(^2)</td>
</tr>
<tr>
<td>Al₂O₃ (AD-94)</td>
<td>2103(^4)</td>
<td>303(^4)</td>
<td>193(^4)</td>
</tr>
<tr>
<td>SiC</td>
<td>2500(^4)</td>
<td>393(^4)</td>
<td>307(^4)</td>
</tr>
</tbody>
</table>

Note: \(^1\) XD Ti-45Al - TiB₂ (HIPed)
\(^2\) Measured
\(^3\) Estimated, based on extrapolation of measured data
\(^4\) Reference [2]

In this study, IMC-reinforced titanium-matrix composites have been produced by powder blending and densifying via high-temperature extrusion deformation processing techniques. Matrix models of composite strengthening predict that the magnitude of strengthening in discontinuously-reinforced ductile materials depends primarily upon the spacing between reinforcing particles. As such, both average reinforcement size, and the magnitude of cross-sectional reduction via extrusion, were varied in an attempt to obtain a range of inter-reinforcement spacings for a constant nominal volume percentage and IMC composition.

The specific goals of the project include:

- determining the extent, if any, of commensurate deformation which occurs between the IMC and the titanium matrix due to high temperature deformation processing,
- characterizing the resulting composite microstructure in terms of the size and aspect ratio of the IMC reinforcement, and their average spacing, and
- correlating these microstructural features to observed mechanical behavior of the composites relative to the unreinforced reference (matrix) alloys.
1. INTRODUCTION

Metal matrix composites have attracted a great amount of attention in the materials science and engineering community in recent years. The past few years have seen an enormous growth and expansion of the technology surrounding the manufacture and application of these materials. The impetus for the development of metal matrix composites is the need for advanced engineering materials with higher-temperature capabilities, high specific strength and specific stiffness and improved environmental resistance. Metal matrix composites are rapidly becoming strong candidates for these kinds of applications as conventional materials that are currently in use reach their performance limits.

Composites are aimed at achieving an enhanced combination of properties by combining at least two chemically dissimilar materials with different properties [3]. By compositing it is possible to develop new materials with unique combinations of properties previously unattainable with conventional materials. Metal matrix composites usually consist of a metal matrix (a metal or alloy), and a reinforcing phase that is usually a ceramic. Various alloys are being used as metal matrices and these include aluminum, copper, titanium, iron and steel, magnesium, nickel and zinc alloys. "Conventional" reinforcements include alumina, boron, boron nitride, boron carbide, titanium boride, silicon carbide, silicon nitride, and graphite. Metal wires such as molybdenum, titanium, stainless steel and tungsten are also being used as reinforcements.
1.1 Classification of Metal Matrix Composites

Metal matrix composites can be classified into three basic types as follows:

(a) Fiber (or whisker) reinforced MMCs, in which the reinforcing phase consists of long continuous fibers or filaments, chopped fibers or whiskers. The diameters of the reinforcing fibers average about 10 to 100 μm, and volume fractions of up to 70% are used. The reinforcing fibers have large aspect ratios (length/diameter).

(b) Particulate reinforced MMCs where the reinforcing phase consists of dispersed particulates or platelets of low length-to-diameter ratios.

(c) Dispersion-strengthened MMCs; the microstructure of these composites consists of a metallic matrix with fine dispersed particles. The dispersoids are hard and insoluble in the matrix and are generally present in small volume fractions.

Each of the classes of MMCs given above is associated with particular characteristics such as processing methods, resultant properties, fabrication costs and applications. Given the classifications of MMCs outlined above, the reinforcements can be alternatively divided into two categories, namely, continuous and discontinuous.

1.2 Continuous Reinforcement of Metal Matrix Composites

Early work on MMCs concentrated on the development of continuous fiber or filament composites for the aerospace and defense industries, where high performance materials were required in relatively low volumes and cost was not a limiting criterion. Considerable research and development resources were expended on the development of continuous carbon and boron fibers which led to the development of aluminum-boron
composites by NASA and the US Department of Defense [4]. However, the high cost of boron fibers, high fabrication costs, and fiber/matrix reactions limited the application of these composites in other industries. The use of continuous fiber reinforced MMCs has expanded into non-aerospace industries in recent years, due primarily to the development of new lower cost fibers. More emphasis is being placed on cheaper reinforcements and rapid and simple production methods, because it is imperative that MMCs should be competitive in cost with alternative engineering materials.

Continuous fiber-reinforced composites are anisotropic; the degree of anisotropy depends mainly on the degree of fiber orientation [3]. The fibers serve as load-carrying components, while the function of the matrix is to hold the fibers together, align them in the desired stress direction, and to transfer and distribute the load to the fibers. The efficiency with which a matrix can transfer the load to the fibers depends on the quality of the fiber/matrix bond. Other roles of the matrix include protecting the fibers from damage, providing environmental protection against, for example, corrosion and oxidation, and isolating fibers so that they do not rub against each other.

The main advantage of continuously-reinforced MMCs is that they have higher specific properties, particularly when considering uni-directional load applications [5]. However, there are disadvantages associated with the use of continuous reinforcements. These include high fabrication costs, fiber/matrix reactions, anisotropic or complex properties (multilayered configurations are required to achieve some degree of isotropy in properties) [3], fiber to fiber contact and fiber damage particularly during fabrication [6].
1.3 DISCONTINUOUS REINFORCEMENT OF METAL MATRIX COMPOSITES

Discontinuous reinforcements include short fibers, whiskers, particulates and platelets. In discontinuously reinforced composites both the matrix and the reinforcement carry substantial portions of the load [5]; unlike in continuously reinforced composites, the matrix strength also plays an important role. Discontinuously-reinforced MMCs can be tailored to yield either isotropic or anisotropic properties depending on the application [4]. One of the main advantages of discontinuously-reinforced MMCs is that they can be processed by conventional metallurgical processes such as casting, extrusion, forging and rolling to produce a wide range of product forms [7]. These fabrication techniques are seriously limited for use with continuous reinforcements. The ease of fabricability and formability of discontinuously reinforced MMCs render them cheaper compared to continuously reinforced MMCs. However, offsetting the processing advantages of discontinuously-reinforced MMCs is the fact that they, generally, have inferior mechanical properties compared to their continuously reinforced counterparts.

1.4 MMC APPLICATIONS AND PERFORMANCE REQUIREMENTS

Metal matrix composites also offer an additional dimension of microstructural tailorability beyond that which can be conventionally achieved through alloying and heat treatment. As a result, numerous combinations of metal matrices and reinforcements are possible in the development of MMCs, thus leading to diverse applications for these materials. Development efforts in the area of MMCs have focussed mainly on increasing strength and stiffness of metal and alloy matrices. Non-mechanical properties (i.e.,
functional) that can be tailored through the use of MMCs include thermal conductivity, coefficient of thermal expansion, and certain magnetic and damping properties.

The major areas of application for MMCs have been the aerospace and defense industries. Other areas of application include the automotive industry, electrical and electronic industries and the leisure markets.

1.4.1 AIRCRAFT AND AEROSPACE APPLICATIONS

Performance demands of aircraft and aerospace systems require the development of new and improved structural materials which are stronger, stiffer, lighter, and they should have elevated temperature capabilities and be able to withstand hostile environments. Areas of application include structural framework, aircraft engines, advanced aerospace projects (such as the U.S. High Speed Civil Plane) and space applications.

The most important requirement in structural aircraft framework is weight reduction. Twenty five percent of unfueled weight of a typical naval aircraft is made up of the airframe [8]. The structural framework must be able to withstand stresses resulting from landings, launch and air maneuvers. Figure 1.1 shows estimated trends in the material constituency of future naval airframe aircraft relative to past implementation. As shown, metal and polymer matrix composites will see increased application in future aircraft; on the other hand, the use of conventional alloys and other structural materials will level off.

Metal matrix composites are also being considered as substitutes for nickel and iron superalloys in aircraft engines. Principal requirements for future aircraft engines are higher thrust to weight ratio, increased fuel efficiency, reduced cost and long service lives [4].
These requirements translate into material requirements of elevated temperature capabilities, increased stiffness, reduced density and increased reliability at higher stress levels. The materials should also have good creep and oxidation resistance. Figure 1.2 shows predicted trends in future aircraft engine material usage.
Specific (density-compensated) properties of MMCs are not as good as those of resin based composites, but they offer a number of advantages over PMCs such as higher temperature capability, improved environmental resistance, higher toughness, and higher thermal and electrical conductivity. Compared to ceramic matrix composites, MMCs have higher toughness and ductility, they are easier to fabricate and are cheaper, but they have inferior elevated-temperature properties. Another advantage of MMCs over CMCs is that the science and technology of metals is much more developed than that of ceramics.

1.4.2 AUTOMOTIVE APPLICATIONS OF METAL MATRIX COMPOSITES

Recent technological challenges facing the automotive industry include the need for improved fuel efficiency, reduced vehicle emissions, enhanced performance, increasing styling options and better quality. Improved efficiency and performance can be achieved by reducing the weight of engine and transmission parts, particularly reciprocating or moving parts [9]. Thus, great opportunities exist for the use of MMCs, particularly discontinuously reinforced MMCs, to meet the challenges outlined above. Efforts are being made to replace conventional materials by light materials such as aluminum, magnesium, titanium, plastics and ceramics. Barriers to the introduction of MMCs and other advanced materials still exist, because each application has to be both technically feasible and cost effective. Cost is a very important factor in the automotive industry because of the need for high volume production. MMCs of interest in the automotive industry have been the more cost effective discontinuously reinforced aluminum composites.

Other advantages offered by the use of MMCs include improved wear resistance, reduced thermal expansion coefficients, higher strength and stiffness and allowing use in more extreme service conditions. Advances made in the production of MMC automobile
parts include the alumina fiber-reinforced aluminum pistons for diesel engines developed by Toyota in 1982 [10] and more recently, alumina and carbon fiber reinforced cylinder liners developed by Honda [11]. Other components that are being developed include brake rotors, rocker arms and calipers [12].

1.4.3 Other Applications of Metal Matrix Composites

The attractive properties of metal matrix composites have made these materials suitable for use in a variety of industrial applications such as wear resistant parts, electrical and electronic components, sporting goods (for example, golf clubs made of aluminum composite), acoustical damping components and thermal management applications [11].

1.5 Summary

Metal matrix composites have garnered great attention from the aerospace, defense, automotive and other industries. Research efforts aimed at addressing performance demands of future advanced materials have led to the development of various processing techniques, new and lower cost reinforcements.

Improved understanding of the reinforcement/matrix interface and how to control its stability still remains as a big challenge. A better understanding of processing/properties relationship will contribute towards reproducibility of properties and hence towards the ultimate success of MMCs.
2. BACKGROUND ON TITANIUM MATRIX COMPOSITES

2.1 INTRODUCTION

Titanium has a unique combination of properties such as high specific strength both at room and moderate temperatures, good fracture behavior and good corrosion resistance [14]. Combining these properties with the high strength and modulus of ceramic reinforcement leads to significant weight savings and improved strength and stiffness. However, titanium is an extremely reactive metal; at the elevated temperatures required for composite fabrication it reacts with ceramic reinforcements to form unwanted reaction products, such as carbides, oxides, silicides and nitrides, which degrade composite properties. These interfacial reaction products are brittle, and usually lead to severe property degradation. It is important to understand the nature of interfacial phenomena in order to design an optimum interface to enhance reliability and service life at elevated temperatures [15].

Advances have been made in the development of titanium-based composites using various combinations of matrices and reinforcements; these will be summarized below.
2.1.1 MATRICES

2.1.1.1 CONVENTIONAL TITANIUM ALLOY MATRICES

Conventional titanium alloys available for MMC development can be divided into four basic classes; (a) lightly alloyed alpha, for example, commercially pure titanium and Ti-5Al-2.5Sn; (b) alpha-beta alloys such as Ti-6Al-4V; (c) the metastable beta alloys, e.g. Ti-10V-2Fe-3Al and (d) stable beta alloys such as Ti-30Mo and Ti-13V-11Cr-3Al. The bulk of titanium-based MMCs under development are based on the Ti-6Al-4V alloy matrix.

2.1.1.2 INTERMETALLIC COMPOUND MATRICES

The elevated temperature capability of titanium based composites can be further enhanced by the use of titanium aluminide intermetallics as matrices, for example, Ti$_3$Al (α$_2$ phase) and TiAl (γ phase). Studies have shown that the performance of these alloys can be further improved by using two-phase "near-γ" alloys that consist of the γ phase with a smaller fraction of the α$_2$ phase. Titanium aluminides offer opportunities for significant weight reductions because of their higher aluminum content compared to conventional titanium alloys. The densities of Ti$_3$Al and TiAl are lower than those of conventional titanium alloys and approximately half that of nickel-base superalloys. The high melting temperatures, good oxidation and creep resistance, and high temperature strength and stiffness of titanium aluminides offer improvements over conventional alloys. Also, titanium aluminides have good modulus retention capabilities at high temperatures because of the strong "A-B" bonding resulting from their ordered structures. The
modulus of commercial titanium alloys, on the other hand, drops rapidly with temperature, from about 96 to 110 GPa at room temperature to about 69 GPa at 540°C [16]. However, titanium aluminides have structurally inherent brittleness at low to moderate temperatures. The low temperature brittleness of these alloys has limited their use even though substantial progress has been made in improving their ductility through the addition of elements such as vanadium, chromium, niobium and manganese. Damage tolerance of intermetallic compounds can be improved by microstructural control and/or by compositing.

2.1.2 REINFORCEMENTS

2.1.2.1 CONTINUOUS REINFORCEMENTS

Fiber reinforcements that have been used in titanium composites include silicon carbide, alumina and boron. One of the major problems with fibrous reinforcements is excessive fiber/matrix reactions and fiber degradation at elevated processing temperatures. These problems result in poor composite properties. Another problem is the development of residual stresses in the matrix and fiber.

Protective fiber coatings have been developed for incorporation in titanium matrices; these include the use of SiC coatings on boron fibers producing the BORSIC fiber, an amorphous B₄C coating for boron filaments and the SCS-6 fiber in which the surface of the stochiometric SiC is graded outwardly to be first carbon-rich and then back slightly towards stochiometry over the last 2 to 3 μm [14]. The SCS-6 fiber has the highest as-manufactured fiber tensile properties compared to the other fibers, hence it has been the most commonly used in titanium matrices.
Continuously reinforced titanium matrix composites are commonly fabricated by solid-state diffusion bonding processes such as vacuum hot pressing or hot isostatic pressing, using fiber preforms, or plasma spraying of fiber-matrix preforms followed by vacuum hot pressing. These fabrication methods are associated with long processing times at elevated temperatures and extensive interfacial reactions. High operating costs, expensive pressing equipment, and vacuum requirements make diffusion bonded composites too expensive for most potential industrial applications [17]. One of the cheaper fabrication techniques for MMCs is liquid infiltration, but the severity of the reaction between titanium matrix and the reinforcing fibers during fabrication has limited its use in the production of titanium matrix composites.

2.1.2.2 DISCONTINUOUS REINFORCEMENTS

Discontinuously-reinforced titanium matrix composites have been developed using particulate or whisker reinforcements. Fabrication methods that have been used to produce discontinuously reinforced titanium matrix composites include in-situ techniques such as plasma processing, self-propagating high temperature synthesis, and powder metallurgy routes. The use of discontinuous reinforcement in titanium matrices presents a big challenge because of the high reactivity of titanium [18]. Efforts to minimize matrix-reinforcement reactions have included the use of low processing temperatures, and/or shorter processing times. These approaches have made it possible to extend the use of discontinuous reinforcements in titanium matrices.
2.2 Interfaces in Titanium Matrix Composites

2.2.1 Interfacial Reactions

The properties of MMCs are largely dependent upon the properties of the interface between the matrix and the reinforcement. The interface is a very important area in the design of viable high-temperature MMCs as a mechanism for load transfer between the matrix and the reinforcement; it is also a possible source of failure during the service life of the composite [19]. It was mentioned in earlier sections that one of the major problems in the fabrication of titanium composites is the occurrence of interfacial reactions. Interfacial reactions occur when reinforcement and matrix components dissociate and inter-diffuse to form new phases or solutions. Most metal matrix composites are thermodynamically non-equilibrium systems, and as such, a chemical potential exists across the reinforcement/matrix interface. Attaining thermodynamic equilibrium involves chemical interaction; the driving force for the chemical interaction is the chemical potential change across the interface. Therefore, at high enough temperatures, during fabrication or in service, diffusion and/or chemical reactions will occur between the components. The interface reaction products generally have properties that are different from those of either the reinforcement or the matrix. Most of the reaction products that form in titanium matrix composites are brittle phases, for example the formation of TiC and Ti$_5$Si$_3$ in titanium-based SiC composites. The presence of brittle phases at the interface degrades the properties of the composites. Interfacial reactions are not entirely undesirable since limited interfacial reactions may enhance bonding and hence load transfer between the components. The extent and products of interfacial reactions will depend on the thermodynamic potential of the elements involved [6].
2.2.2 INTERFACIAL STABILITY

The degree to which an interface of a metal matrix composite can maintain stability will determine to a large extent, its success as a high temperature material.

Metcalfe [20] proposed the following general classification of types of interfaces:

Class I: Reinforcement and matrix mutually non-reactive and insoluble.
Class II: Reinforcement and matrix mutually non-reactive but soluble.
Class III: Reinforcement and matrix react to form at least one compound at the interface.

According to the classification given above, a Class I interface can be characterized by a stable reinforcement/matrix interface; on the other hand, a Class III interface is characteristic of an unstable reinforcement/matrix interface. Titanium matrix composites are mainly of Class III; since these composites are being considered for high temperature applications it is important therefore to define ways of controlling their interfacial stability. Another problem that may arise due to the instability of the interface is void formation by the Kirkendall effect. The voids result from an imbalance in the species transported across the interface. The presence of residual porosity at the original interface between the matrix and the reinforcement can accelerate the nucleation of voids [20]. Volume change during formation of interfacial products can also contribute to void formation.

2.2.3 THERMODYNAMICS AND KINETICS OF INTERFACIAL REACTIONS

An understanding of the thermodynamics and kinetics of composite interfacial reactions is important in designing metal matrix composites for high-temperature applications. Thermodynamic phase-equilibria data can help in identifying matrix and reinforcement materials that may result in stable interfaces, but they do not tell us what combinations of
phases may form in the process of achieving equilibrium [21]. Reaction kinetics data can be used to estimate the rate and the extent of any reaction that may occur at the interface.

Interfacial reaction kinetics studies have been done on several titanium composites, particularly fiber-reinforced composites. The studies usually involve the measurement of interfacial layer thickness at different temperatures for different times. The thickness of the interfacial layer is a measure of the extent of the reaction. If a linear relationship between the reaction layer thickness and time at a planar interface exists, the reaction kinetics are interface-controlled [19]. This kind of relationship means that the reaction rate does not depend on the thickness of the interfacial layer but it depends on some process occurring at the interface, for example, dissociation of the reinforcement or combining of the reinforcement and the matrix to form a new compound. If the thickness of the interfacial zone at a planar interface changes with the square root of time, i.e

\[ x = k \sqrt{t} \]  

(2.1)

where \( x \) is the reaction layer thickness, \( k \) the rate constant and \( t \) the time, then growth is controlled by the diffusion of one of the reacting species through an interfacial zone of constant interfacial area and constant boundary conditions. The constant \( k \) has the dimensions of a diffusion coefficient, \( \text{L}^2\text{T}^{-1} \). Equation (1) assumes that the reaction rate constant, \( k \), is temperature dependent, and its dependence can be represented by an Arrhenius equation [20]

\[ k = k_0 e^{\frac{-Q}{RT}} \]

(2.2)

where \( k_0 \) and \( Q \), are the pre-exponential constant and activation energy, respectively, and are material constants; \( T \) is the absolute temperature and \( R \) is the gas constant. Equation
(2.1) has been found to describe adequately most of the growth data in fiber reinforced matrices of titanium and its alloys.

Several reaction kinetics studies on titanium-based composites continuously reinforced with ceramic fibers such as boron, SiC and alumina have been carried out. On the other hand, reaction kinetics studies on discontinuously reinforced titanium composites are very limited. Studies done on boron fiber-reinforced Ti-8Al-1Mo-1V alloy found that TiB$_2$ was the only reaction product formed [20]. The experiments covered the temperature range 750 to 1000°C for times up to 200 hours; the thickness of titanium diboride formed varied from about 1 to 9μm and the reaction kinetics followed the parabolic rate equation. Martineau et al. [22] studied the interfacial reactions between SiC-based CVD-filaments and titanium matrices (commercially pure titanium and Ti-6Al-4V). They found that the growth of the fiber/matrix reaction layer was mainly diffusion-controlled, thus the rate of formation of the interfacial zone increased rapidly with increasing temperature. Martineau et al. also found that, at a given temperature, the growth rate of the fiber/matrix reaction zone is lower for alloyed Ti-6Al-4V matrix than for unalloyed titanium.

Some of the microstructural changes that are associated with the formation of thick interfacial reactions include:

(i) in the thick reaction layers one of the reaction product sublayers could become partly disconnected from the others due to pore precipitation,

(ii) each filament becomes progressively consumed, thus the surface through which diffusion occurs becomes smaller,

(iii) the matrix becomes saturated in the diffusing species.
2.2.4 CONTROL OF INTERFACIAL REACTIONS

Several approaches are possible for reducing reinforcement/matrix interactions. They include:

(a) The use of short processing times to minimize the extent of detrimental reactions. For example, Warrier and Lin used rapid infrared forming (RIF) to fabricate titanium composites reinforced with SiC fibers [17]. This technique involves very short processing times, 1 to 2 minutes, and cooling rates of up to 200°C/s during fabrication are possible. These researchers report that RIF resulted in composites with limited and controlled interfacial reactions with mechanical properties that were either comparable or superior to those of titanium composites produced using the diffusion-bonding techniques.

(b) Reduction of processing temperatures to minimize reactions. Since the interfacial reaction rate is thermally activated, a decrease in the fabrication temperature can affect the reaction kinetics.

(c) Use of coatings to prevent adverse chemical interactions, improve wettability and adhesion, to enhance bonding between the reinforcement and the matrix. Coatings must be compatible with both the matrix and the reinforcement. However, there are difficulties associated with the use of coatings, such as the lack of thermodynamic and kinetic data to use in choosing appropriate coatings, and the inability to produce defect-free and uniform coatings on the reinforcements [21].

(d) Reduction of diffusion rates by using alloying elements. This effect has been achieved by, for example, alloying titanium with slower diffusing elements to reduce interfacial reactions. In studies done on Ti matrices reinforced with boron fibers, it was found that vanadium, molybdenum and aluminum caused significant reductions in reaction kinetics, whereas tin, silicon, germanium and copper had little effect [20]. The study suggested that the optimum matrix would contain enough diboride modifier, such as vanadium, to
reduce boron transport; as well as additions that reduce the activity of titanium, such as molybdenum or aluminum.

Interfacial reactions in titanium composites can be minimized by using reinforcements that are chemically compatible with the matrix. In order to accomplish this, reinforcements need to be customized specifically for titanium matrices, either through interfacial modification of existing fibers or by creation of new families of reinforcements.

2.3 IN-SITU PROCESSING OF TITANIUM MATRIX COMPOSITES

2.3.1 INTRODUCTION

In-situ composites are materials in which the reinforcement is formed "on site" directly within the matrix to be reinforced. Since in situ processing techniques yield thermodynamically-compatible reinforcements, they result in composites which are essentially free of any detrimental chemical reactions between the matrix and the reinforcement. One of the main advantages of in-situ processing techniques is the absence of externally-derived contaminants on the reinforcement surface (e.g. those from oxidation), which might otherwise be introduced during processing or handling. The clean interfaces resulting from in-situ processing result in good bonding between the matrix and the reinforcement; this is good for MMCs where strength is the main concern. Also, since the reinforcements are generated in-situ, the safety hazards associated with handling of fine metal or ceramic particles or whiskers are avoided [4]. In-situ processing techniques also potentially lower the cost of producing metal matrix composites.

The term in-situ was originally used to refer to composites produced by the directional solidification of polyphase eutectic alloys, but it is now used to refer to several processing techniques. In-situ processing strategies can be divided into two broad categories:
constitutional strategies, and morphological strategies.

2.3.1.1 Constitutional strategies

In constitutional in situ processing, the reinforcement is directly created in a metal matrix using methods where its precipitation and growth can be carefully and creatively controlled. Examples include processing techniques such as:

(a) Liquid-gas reactions. In these reactions the reinforcing phase is the product of a gas-liquid reaction resulting from the injection of a gas into a reactive liquid metal. A rapid reaction that occurs between the solute alloying elements and the gas introduced into the matrix produces a fine dispersion of the reinforcement in the matrix alloy [23]. This technique has been used to form carbides and nitrides in aluminum, copper, nickel and intermetallic matrix composites.

(b) Solid-gas (reactive plasma). This processing method utilizes high thermal energies of a plasma to create reactive chemical species in the presence of heated vapor, liquid, and/or solid [23]. Solids precipitate from the gas.

(c) Liquid-solid processing (reaction synthesis). An example of a liquid-solid reaction processing technique is the XD™ developed by Martin Marietta Corporation. This technique is used to make in-situ ceramic particle-reinforced composites. Powders of the elemental components of the reinforcing phase are mixed or incorporated into the metallic or intermetallic matrix material. The composite mixture is then heated to a high temperature, to a point where the elemental constituents react exothermically to completion by way of a self-sustaining reaction. The reinforcing phase is precipitated in the matrix solvent medium via diffusion of the components; this reaction results in the formation of a dispersion of reinforcing particles in the matrix.
2.3.1.2 MORPHOLOGICAL STRATEGIES

In morphological in-situ processing the composite microstructure is produced through creative processing of an otherwise "conventional" multiphase alloy. Examples of morphological in-situ strategies include:

(a) **Directional solidification.** This technique involves controlled unidirectional solidification of a liquid eutectic alloy. A metal matrix composite with either continuous fibrous or lamellar microstructure is produced [24].

(b) **Lanxide process.** Two processes for making metal-matrix composites were developed by the Lanxide Corporation for producing metal matrix composites from liquid metal alloys. (i) The DIMOX or directed metal "oxidation" process in which controlled oxidation of a liquid metal alloy such as Al-Si-Mg produces a monolithic metal/ceramic matrix with fiber and/or particulate reinforcement. (ii) The PRIMEX process which involves spontaneous (pressureless) infiltration of filler preforms with molten metal through a preform of the reinforcing phase [25]. The Lanxide process is typically used to produce near net shape parts.

(c) **Deformation processing.** The composite is formed during mechanical processing, that is, during rolling, extrusion, wire drawing or swaging of a multi-phase billet. The phases codeform, and the resultant morphology consists of an elongated minor phase within the matrix.

2.3.2. REACTION SYNTHESIS OF INTERMETALLIC MATRIX COMPOSITES

Reactive synthesis techniques have been used to produce intermetallic and ceramic compounds from reactions between elemental constituents. The initial reactants are ignited and they react to form the constituents of the composites by way of a self-
sustaining, exothermic reaction. The driving force for the reaction is high thermodynamic stability of the products [26]. Hence this process produces thermodynamically stable composites with high reaction kinetics. The composites produced have fine reinforcements, and high reinforcement volume fractions can be produced. This processing technique can be used as a primary processing method, which results in a highly porous product. Conventional metallurgical processing is used to obtain full density and/or the required shape. This processing technique can be used to produce TiB$_2$-reinforced TiAl intermetallic composites (IMCs). Reactive synthesis has been used to produce various carbides, borides, silicides, nitrides, hydride and oxide-reinforced metal matrix composites.

### 2.3.3 Deformation Processing of Metal Matrix Composites

Deformation processing can be used as a secondary processing technique to form in-situ composites. A primary fabrication technique such as casting or powder processing is used to produce a two-phase material. The two-phase material is then deformation processed by extrusion, rolling, swaging or wire drawing to align the reinforcing phase and consolidate the product.

#### 2.3.3.1 Extrusion of Composite Powders

Extrusion offers opportunities for fabricating composites using lower processing temperatures compared to fusion metallurgy techniques, and a wider capability for making in-situ composites [27]. There are two types of extrusion, namely, direct and indirect extrusion. In direct extrusion the ram pushes a workpiece forward through a die, thus reducing the cross-sectional area of the workpiece. In indirect extrusion the workpiece
remains stationary relative to the container. Direct extrusion is more commonly used than indirect extrusion.

Canned extrusions are used to fabricate in-situ composites; the reinforcing phase may be introduced during powder blending or it may form in-situ. The extrusion can is evacuated and sealed so that pores can close during extrusion. The composite powder mixture is then consolidated at high temperature.

2.3.3.2 MECHANICS OF POWDER EXTRUSION

An allowable range of processing conditions during extrusion is defined by the relationship between pressure and temperature on the deformation level on one hand, and the relationship between strain rate and temperature on allowable deformation level on the other hand. The extrusion pressure depends on the flow stress of the materials, temperature, die or can friction, strain rate and the extrusion ratio. When a reinforced metal matrix composite mixture is extruded the areas containing the reinforcing particles act as hard regions therefore strain partitioning takes place during deformation. At low strain rates the deformation is accommodated mainly by the particle-free regions. The strain-rate sensitivity of the matrix is higher than that of the regions with particles, therefore when a high strain rate is applied, the matrix forces deformation to occur in the particle regions leading to their shearing. Successful co-reduction of the different phases depends on their thermomechanical and metallurgical compatibility. High extrusion ratios are required for complete alignment of the reinforcement and breaking up of prior particle boundaries in order to obtain improved mechanical properties [27].
2.4 Strengthening in Discontinuously Reinforced Metal Matrix Composites

Some of the models that have been used to explain composite strength include rule of mixture-based (ROM) models and Orowan theory. ROM models fail to explain the strengthening in discontinuously reinforced metal matrix composites. On the other hand, the Orowan theory underestimates the strengths of discontinuously-reinforced composites when the reinforcing phase consists of large particles or the spacings between particles are large [28]. Models that explain the strength in terms of matrix-based models give better estimates of strengths of particulate-reinforced metal matrix composites. The presence of second-phase particles in the continuous matrix results in localized internal stresses which modify the plastic behavior of the matrix [29]; thus the strengthening is due to microstructural changes in the matrix. Factors that contribute to the strengthening from second phase particles include size, shape and distribution of the reinforcing phase, the strength, ductility and strain behavior of the matrix and the reinforcement, the interfacial energy and interfacial bonding between the phases.

During deformation of the composite, the dislocation density increases in the vicinity of the reinforcing particles [29]. The applied stress, $\sigma$, required to deform through a field of particle-stabilized dislocations can be represented by the following equation:

$$\sigma = \sqrt{\frac{G_m E_p b}{\lambda}}$$  \hspace{1cm} (2.3)

where $G_m$ is the shear modulus of the matrix, $b$ is the matrix Burger's vector, $E_p$ is the elastic modulus of the reinforcement, and $\lambda$ is the spacing between the reinforcing particles. It can be seen from equation (2.3) that the stress required to deform a particle-reinforced matrix increases with decreasing spacing between the particles.
Since the origin of strengthening also relies upon the dislocation density in the material, an alternative equation describing the flow strength of the composite is

\[
\sigma_{\text{flow}} = \sigma_o + \alpha G_m b \sqrt{\rho}
\]  

(2.4)

where, \( \sigma_o \) is the flow stress at zero plastic strain, \( \alpha \) is a constant with values ranging between 0.1 and 1.5 and \( \rho \) is the dislocation density of geometrically necessary dislocations due to the presence of the non-deforming reinforcing particle [30, 31]. The density of geometrically necessary dislocations is related to the plastic strain, \( \varepsilon_p \), and interparticle spacing according to [29-31]

\[
\rho \approx \frac{\varepsilon_p}{\lambda_e}
\]  

(2.5)

Equations (2.4) and (2.5) can be combined to give the dependence of flow stress on interparticle spacing, i.e

\[
\sigma_{\text{flow}} = \sigma_o + \alpha G_m b \sqrt{\frac{\varepsilon_p}{\lambda_e}}
\]

(2.6)

the other variables have been explained. As with equation (2.3), equation (2.4) predicts that the flow stress of the particle-reinforced composite increases with decreasing spacing between reinforcing particles.
2.5 **SUMMARY**

The key points pertinent to the present thesis which have been addressed in this chapter can be summarized as follows:

1. Metal matrix composites have potential as materials of the future but are currently difficult to process because of the high reactivity of metal matrices.

2. The reactivity of metal matrices leads to the formation of undesirable interfacial reactions between the matrix and the reinforcement. Interfacial reactions lead to composite property degradation.

3. In-situ approaches reduce reactivity problems and they result in composites with clean interfaces.

4. Deformation processing has been shown to be capable of evolving composites with high strengths. Deformation processed composites exhibit strengths that are in excess of those predicted by rule of mixture (ROM) models.

5. Strengthening in particle-reinforced composites increases with decreasing spacing between the reinforcing particles.

6. Intermetallic matrix composites offer new possibilities as reinforcements for metal matrices. IMCs can be created to exhibit ceramic-like behavior at ambient-temperatures (i.e. high strengths) and metal-like behavior at high temperatures (i.e. deformation processable).
3. EXPERIMENTAL PROCEDURES

Commercially pure titanium (CP Ti) matrix composites were produced using particulate TiB₂-reinforced near-gamma titanium aluminide as the reinforcing phase. The first step in the fabrication of the composites was the reaction synthesis of the near-γ TiAl intermetallic matrix composite(IMC) reinforcement. The as-synthesized IMC sponge was reduced into powder and blended with commercially pure titanium powder. The composite powder mixture was deformation processed high temperature extrusion. An overview of the processing approach used is shown in Figure 3.1.

Figure 3.1: An overview of the processing used to make IMC-reinforced titanium matrix composites.
3.1 SYNTHESIS OF \((\text{IMC})_p\) REINFORCEMENT

3.1.1 FORMULATION AND REACTION SYNTHESIS OF IMC

Reaction synthesis was used to produce TiB₂-reinforced near-\(\gamma\) TiAl IMC. Elemental aluminum, boron and commercially pure titanium powders were used as reactants in the reaction to form the IMC. Elemental aluminum powder of \(-100\) mesh size and 99.97% purity was obtained from CERAC, Inc. (Milwaukee, Wisconsin). Crystalline elemental boron powder was also obtained from CERAC; the powder was 99.5% pure and of \(-100\) mesh size. Commercially pure, dehydrated elemental titanium powder was obtained from Micron Metals, Inc. (Salt Lake City, Utah). The titanium powder had a 99.4% minimum purity level, and it was of \(-100\) mesh size range.

The precursor elemental powders were mixed in proportion to yield a nominal composition of Ti-46 at%Al + 20 vol% TiB₂. Table 3.1 gives weight percentages used in the formulation of the IMC.

The powder mixture was sent to Martin Marietta Laboratories (Baltimore, MD) where it was blended and mechanically pressed into \(\approx 101 \times 101 \times 6\) mm wafers. The green compacts were synthesized into IMC utilizing induction heating in an evacuated vacuum chamber.

<table>
<thead>
<tr>
<th>Material</th>
<th>Weight percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium</td>
<td>67.88</td>
</tr>
<tr>
<td>Boron</td>
<td>7.08</td>
</tr>
<tr>
<td>Aluminum</td>
<td>25.04</td>
</tr>
</tbody>
</table>
3.1.2 COMMINUTION AND SIZING OF IMC

The IMC sponge resulting from the reaction synthesis reaction was reduced into powder by first using a jaw crusher to form IMC chunks. The IMC chunks were subsequently reduced into powder using a disk mill and/or mechanical attrition at XFORM, Inc. (Cohes, New York).

Sieving of the milled IMC powders was carried out to separate the powders into size distributions required for composite formulation. Two IMC powder size distributions, -50/+120 (IMC1) and -200/+325 (IMC2), were obtained.

3.2 PROCESSING OF COMPOSITES

3.2.1 FORMULATION OF METAL MATRIX COMPOSITES

Each IMC powder distribution was mixed and blended with the -325 mesh CP titanium from to yield a nominal composite composition of Ti + 20 vol% IMC. Calculations for the proportions of the composite components were based on theoretical and the measured apparent densities of CP titanium and the IMC composites. Table 3.2 gives the formulations of the composites.

<table>
<thead>
<tr>
<th>Component</th>
<th>Nominal volume</th>
<th>Actual weight</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>percent</td>
<td>percent</td>
</tr>
<tr>
<td>IMC</td>
<td>20</td>
<td>18.4</td>
</tr>
<tr>
<td>CP Ti</td>
<td>80</td>
<td>81.6</td>
</tr>
</tbody>
</table>

Table 3.2: Composite formulation
3.2.2 Extrusion of Powders

CP titanium pipe was used to make extrusion cans for the extrusion of the composites. The extrusion cans were 15 cm long with an outside diameter of 7.3 cm. Nose and tail sections were welded onto the extrusion cans at Martin Marietta Laboratories. Extrusion was performed by personnel of Universal Energy Systems, Inc. at Wright Patterson Air Force Base (Dayton, Ohio). The composite powder mixtures were blended, poured into the extrusion cans and hermetically sealed under a vacuum of about $10^{-3}$ atm. The billets were held at 1066°C for two hours, removed from the furnace, and immediately extruded. Two extrusion die ratios were used, 14:1, for two of the composites, and 19.8:1 for a third composite. Less than six seconds elapsed from the moment the billet was removed from the furnace to completion of extrusions. The extruded composites were slow-cooled at a rate of about 0.1°C/s to room temperature in vermiculite. As a reference alloy, an extrusion containing the matrix only (CP Ti) was performed at an extrusion of 14.3:1. The processing variables, that is, two powder size distributions and two extrusion ratios, yielded the following as-processed materials:

<table>
<thead>
<tr>
<th>Material</th>
<th>IMC powder size</th>
<th>Die ratio</th>
<th>Final extrusion ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td></td>
<td>14.3:1</td>
<td>14.7:1</td>
</tr>
<tr>
<td>CPLR</td>
<td>-50/+120 mesh</td>
<td>14:1</td>
<td>14.5:1</td>
</tr>
<tr>
<td>FPLR</td>
<td>-200 mesh</td>
<td>14:1</td>
<td>14.4:1</td>
</tr>
<tr>
<td>CPHR</td>
<td>-50/+120 mesh</td>
<td>19.8:1</td>
<td>19.8:1</td>
</tr>
</tbody>
</table>
In Table 3.3, CPLR refers to the composite formulated with coarse IMC powder and low extrusion ratio. FPLR refers to the composite formulated with fine IMC powder and a low extrusion ratio. CPHR is the composite formulated with coarse IMC powder and a high extrusion ratio.

Portions of the extruded matrix and two of the three composites were sent to Howmet Opperhall Research Center (Whitehall, Michigan) for hot isostatic pressing (HIP'ing) to remove possible porosity remaining after extrusion. HIP'ing was done at 1260°C and 207 MPa for 4 hours.

3.3 IMC CONSOLIDATION

IMC (IMC1 and IMC2) powders were consolidated using HIP'ing, in an attempt to characterize the "monolithic" properties of the IMC. Powder samples were placed in pyrex tubes and evacuated. The tubes were then sealed and HIP'ed according to the cycle given in Table 3.4. Segment 1 of the HIP cycle served to soften and collapse the glass tube around the enclosed sample. Once collapsed, the temperature and pressure were simultaneously increased (segments 2 - 4) to effect consolidation.
Table 3.4: HIP cycle used in the consolidation of IMC powders

<table>
<thead>
<tr>
<th>Segment number</th>
<th>Temperature (°C)</th>
<th>Pressure (psi)</th>
<th>Segment duration (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>700</td>
<td>15</td>
<td>90</td>
</tr>
<tr>
<td>2</td>
<td>900</td>
<td>12771</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>1225</td>
<td>24656</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>1225</td>
<td>24656</td>
<td>120</td>
</tr>
<tr>
<td>5</td>
<td>20</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

3.4 CHARACTERIZATION OF IMC

3.4.1 CHARACTERIZATION OF AS-SYNTHESIZED IMC AND CONSOLIDATED IMC

For the characterization of the as-reacted IMC, optical and scanning electron microscopy were used. Metallographic observation was performed on lightly etched samples; H₂O + 2 vol% HF was used as the etchant. TiB₂ particle sizes within the IMC were measured by direct measurement on optical and SEM micrographs using a hand-held magnifying (7X) eyepiece.

The consolidated IMC samples were similarly examined using optical microscopy.
3.4.2 IMC POWDER SIZE DISTRIBUTION MEASUREMENTS

The size distribution of milled and subsequently sieved IMC powders were measured using both manual measurements on SEM micrographs, and a laser scattering particle size distribution analyzer.

3.5 CHARACTERIZATION OF EXTRUDED COMPOSITES

Extruded composites were characterized using optical microscopy and a scanning electron microscope interfaced with an energy dispersive X-ray microanalysis (EDX/EDS). All the observations were done on lightly etched samples (H$_2$O + 2 vol% HF) cut parallel to the extrusion direction. Two of the composites and the matrix were characterized both in the as-extruded and the as-extruded and HIP'ed conditions.

SEM micrographs were used to measure IMC particle size, center-to-center spacing between the IMC particles and the thicknesses of the interfacial layers. The center-to-center spacings between IMC particles were determined using the linear intercept method.

The minor and major diameters were measured for each particulate. The diameters of the particulates are reported as the equivalent diameter of a sphere of equal volume to the ellipsoid shape measured. The minor and major diameters were also used to calculate the particle aspect ratios, $\frac{d_{\text{max}}}{d_{\text{min}}}$. Edge-to-edge interparticle spacings were determined by subtracting twice the statistically corrected ($r^*=0.816r$) mean particle radius. The volume fraction of the reinforcing phase was measured off the SEM screen, using the point count method.

Interfacial sublayers were measured by direct measurements on SEM micrographs. At least ten measurements of the interfacial sublayers were made per reinforcement particle.
Energy dispersive X-ray semi-quantitative microanalysis was used to determine aluminum and titanium compositional change throughout the microstructure using the spot mode. The distance of the spot from the particle/matrix interface was measured and the composition profile of each spot was plotted (X-ray peak intensity vs K-α energy value).

3.6 EVALUATION OF MECHANICAL PROPERTIES

3.6.1 MICROHARDNESS

Vickers microhardness measurements were performed on the matrix and the composites, using both as-extruded and HIPed samples. The samples were polished and etched to enable distinction between the phases and interfacial sublayers; separate measurements were taken for each feature (particle, matrix and interfacial layers). A minimum of ten measurements was taken for each feature measured. A Leco DM 400 microhardness tester was used with a 300 g·f indentation load for the measurements.

3.6.2 COMPRESSION TESTS ON COMPOSITES AND IMCS

Compression test specimens of the composites were prepared from the specimens used in the tensile tests. All the specimens were cut and ground to have height/diameter ratios of about 1.75 in order to avoid edge effects or buckling during compression testing. IMC compression specimens were prepared from the as hot isostatically pressed samples. All the compression tests were performed using an Instron 4206 machine fitted with round plates. All the measurements were done at room temperature at an initial strain rate of \(1 \times 10^{-4} \text{s}^{-1}\).
3.6.3 TENSILE TESTING OF MATRIX AND COMPOSITES

Hot isostatically pressed longitudinal sections of the extruded matrix and composites were used to machine tensile specimens. Cylindrical tensile specimens with a gauge diameter of 6mm and gauge length of 40mm were machined at Martin Marietta Laboratories. Figure 3.2 is a schematic representation of the tensile specimen used. All the tensile tests were performed at room temperature and at a strain rate of $7.2 \times 10^{-3}$ s$^{-1}$, using an Instron 4206 testing machine.

![Diagram of tensile specimen]

Figure 3.2: A schematic representation of a tensile specimen.
4. EXPERIMENTAL RESULTS

4.1 MICROSTRUCTURE

4.1.1 MICROSTRUCTURE AND CHARACTERIZATION OF IMC

4.1.1.1 General features

The near-γ TiAl IMC, with nominal composition Ti-46Al + 20 vol% TiB₂, was produced using reactive synthesis and is shown in the as-synthesized form in Figs 4.1 and 4.2.

The microstructure of the IMC consists of fine TiB₂ particulates segregated to boundary regions within the microstructure. A smaller fraction of the TiB₂ particulates reside within the matrix grains. The matrix exhibits a lamellar morphology consisting of α₂ (Ti₃Al) and γ (TiAl) laths. The IMC microstructure also contained large carbide particles and pores. The carbides are believed to be the result of contamination from the graphite crucibles used during synthesis. SEM micrographs also revealed cracks in the IMC.

4.1.1.2 TiB₂ PARTICLE SIZE MEASUREMENTS

The size of TiB₂ particles within the IMC was determined by direct measurement on optical and SEM micrographs. Since the particles were very fine, only one dimension was measured. Particle size distribution is given in Figure 4.3.

The mean TiB₂ particle size was 0.755 μm, the median was 0.667 μm and the standard deviation was 0.3404 μm.
Figure 4.1: Optical micrographs of as-synthesized Ti-46Al + 20 vol% TiB$_2$: (a) taken on a polished surface, it shows TiB$_2$ particles, large carbide particles (explained in the text) and porosity; and (b) taken on an etched surface (note the lamellar morphology of the two phase matrix).
Figure 4.2: An SEM micrograph of as-synthesized IMC showing the segregation of TiB$_2$ particles, seen to be mostly at grain boundaries.

Figure 4.3 TiB$_2$ particle size distribution in IMC
4.1.1.3 COMMINUTION AND SIZING OF IMC

The powder size distribution of the milled IMC sponge is given in Table 4.1.

In order to determine the effect of IMC precursor powder size on composite strength, two powder size distributions were used in the formulation of the composites. IMC1 consisted of -50/+120* mesh size powder, and IMC2 of a mixture of 43 wt% of -200/+325 and 57 wt% of the -325 mesh powder. The size and shape of IMC1 and IMC2 powders are shown in Figure 4.4.

IMC powder size measurements were done for each sieve distribution IMC using both manual measurements on SEM micrographs, and a laser scattering particle size distribution analyzer. For the latter, two samples of each powder were analyzed using a 5M aqueous solution of Calgon® as a scattering medium. Laser scattering particle size analysis results are shown in Figure 4.5.

<table>
<thead>
<tr>
<th>Mesh size</th>
<th>Sieve opening size (μm)</th>
<th>Weight percent retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>+50</td>
<td>&gt;300</td>
<td>26.56</td>
</tr>
<tr>
<td>-50/+120</td>
<td>&gt;125</td>
<td>35.77</td>
</tr>
<tr>
<td>-120/+200</td>
<td>&gt;75</td>
<td>15.09</td>
</tr>
<tr>
<td>-200/+325</td>
<td>&gt;45</td>
<td>9.7</td>
</tr>
<tr>
<td>-325</td>
<td>&lt;45</td>
<td>12.5</td>
</tr>
</tbody>
</table>

* Refers to powder which passes through a 50 sizing screen but which is retained on a size 120 screen.
Figure 4.4: The size and shape of (a) IMC1 (-50/+120 mesh size) and (b) IMC2 (-200 mesh size).
A summary of the particle size analyses is given in Table 4.2.

<table>
<thead>
<tr>
<th></th>
<th>Manual measurements</th>
<th>Electronic measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td>IMC1 Mean(μm)</td>
<td>195.75</td>
<td>122.2</td>
</tr>
<tr>
<td>IMC1 Median(μm)</td>
<td>201.48</td>
<td>113.7</td>
</tr>
<tr>
<td>IMC1 Standard deviation</td>
<td>91.98</td>
<td></td>
</tr>
<tr>
<td>IMC2 Mean</td>
<td>15.28</td>
<td>45.86</td>
</tr>
<tr>
<td>IMC2 Median</td>
<td>12.76</td>
<td>45.01</td>
</tr>
<tr>
<td>IMC2 Standard deviation</td>
<td>12.9</td>
<td></td>
</tr>
</tbody>
</table>

Manual and electronic measurements gave significantly different results; in the case of IMC1 manual measurements gave larger powder particle size. The average powder size of IMC2 obtained by electronic measurement is about three times that obtained through manual measurements. Possible causes of the discrepancy between the two measurement methods are: (i) if there is multiple scattering from powder particles, their size appears small to the laser analyser, (ii) particles tend to orient as they pass through the sensor, thus the measurement would be skewed towards the preferred orientation, (iii) coagulation of particles during measurement skews particle measurement towards larger sizes.

The lower IMC1 particle size measured by the laser analyzer is probably due to points (i) and (ii). Particle coagulation probably resulted in larger IMC2 particle size measurement by the laser analyzer.
Figure 4.5: IMC particle size analysis data: (a) IMC1(-50/+120 mesh) and (b) IMC2(-200 mesh).
4.1.1.4 X-ray analysis of IMC

An X-ray pattern of the IMC powder is given in Figure 4.6.

![X-ray diffraction pattern of IMC (Ti-46Al + 20 vol% TiB2)](image)

As expected, the main X-ray peaks identified were those of TiAl (γ), Ti₅Al (α₂) and TiB₂. Trace amounts of the TiAl₃ and Ti₅B₄ phases were also identified. While the IMC micrographs showed the presence of carbides (Fig. 4.1), they were not detected during X-ray analysis.

4.1.1.5 Consolidation of IMC powders

In an attempt to characterize the "monolithic" properties of the IMC, samples were consolidated by hot isostatic pressing (HIP). The IMC was well consolidated after hot isostatic pressing as shown in Figure 4.7.
Figure 4.7: An optical micrograph of HIP'ed IMC (note the lamellar morphology of the near-$\gamma$ TiAl matrix and the TiB$_2$ particles).
4.1.2 Matrix Particle Size Measurements

Commercially pure titanium of mesh size -325 was blended with the IMC powders to make the IMC-reinforced metal matrix composites. Figure 4.8 is an SEM micrograph of the unblended titanium powder.

Figure 4.8: The size and shape of the CP titanium powder used as the matrix constituent in the composites.
The Ti sponge consists of irregularly shaped particles. The powder size and its distribution were measured using the laser scattering particle size distribution analyzer; the results are shown in Figure 4.9. Particle size analysis of matrix powder gave a mean particle size of 32.56 μm and a median of 31.55 μm. Manual measurements of mean Ti particle size were not performed.

Figure 4.9: Particle size distribution of the titanium matrix powder.
4.1.3 Characterization of Extruded Matrix and Composites

4.1.3.1 General Microstructural Features

The reinforcement (IMC) and the matrix (CP Ti) powders were blended to yield composite powder mixtures with a nominal composition of Ti + 20 vol% IMC (i.e. Ti-46Al + 20 vol% TiB₂). IMC powder particle size and extrusion ratio were used in an attempt to effect different resultant IMC spacings in the final composites. Table 4.3 gives the different variables used in composite processing.

<table>
<thead>
<tr>
<th>Material</th>
<th>IMC powder size</th>
<th>Die ratio</th>
<th>Final extrusion ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td></td>
<td>14.3:1</td>
<td>14.7:1</td>
</tr>
<tr>
<td>CPLR</td>
<td>-50/+120 mesh</td>
<td>14:1</td>
<td>14.5:1</td>
</tr>
<tr>
<td>FPLR</td>
<td>-200 mesh</td>
<td>14:1</td>
<td>14.4:1</td>
</tr>
<tr>
<td>CPHR</td>
<td>-50/+120 mesh</td>
<td>19.8:1</td>
<td>19.8:1</td>
</tr>
</tbody>
</table>

As noted previously, CPLR refers to the composite formulated with coarse IMC powder and low extrusion ratio. FPLR refers to the composite formulated with fine IMC powder and a low extrusion ratio. CPHR is the composite formulated with coarse IMC powder and a high extrusion ratio.

All the extrusions were outwardly successful with no macroscopic cracking or splitting observed. Figure 4.10 is an optical micrograph of an extruded and subsequently HIP'ed matrix. The matrix was well consolidated, with no significant porosity observed.

The microstructures of composites CPLR and FPLR were evaluated in both the as-extruded and extruded + HIP'ed conditions (CPHR was not HIP'ed). The microstructures of the as-extruded composites are shown in Figures 4.11, 4.12 and 4.13.
Figure 4.10: An optical micrograph of extruded and HIP'ed matrix.

Figure 4.11: An SEM micrograph of a longitudinal section of composite CPLR in the as-extruded condition (extrusion direction is horizontal). The matrix flows around the reinforcement, showing that most of the extrusion derived deformation has been accommodated by the lower flow strength matrix.
Figure 4.12: An SEM micrograph of a composite FPLR in the as-extruded condition (extrusion direction is horizontal). The micrograph shows a significant amount of inter-diffusion between composite constituents.

Figure 4.13: A longitudinal section of composite CPHR in the as-extruded condition (extrusion direction is vertical). Note the matrix envelope around the reinforcement particle.
The micrographs of CPLR and CPHR, Figure 4.11 and 4.13, show that the deformation imposed during extrusion of the composite powder blend was accommodated by the matrix component only (this feature of the microstructure is not clearly obvious in FPLR). The IMC reinforcing phase exhibited very limited deformation. The deformation-processing was not able to yield the desired composite microstructure of elongated short fibers of IMC reinforcement as illustrated in Fig. 3.1. This is probably due to the fact that the ductile matrix was not able to transfer the deformation load to the reinforcing phase.

The micrographs also show porosity within and around the IMC particle reinforcement. The porosity within the IMC is mainly due to the initial porous nature of the IMC. Additional voids are present around the reinforcement due to inter-diffusion between the composite constituents and the presence of interfacial reaction layers between the matrix and the reinforcement. The presence of voids is more pronounced in FPLR (fine powder, low extrusion ratio) than in the other two composites. The reaction layers are more clearly defined in composites CPLR (coarse powder, low extrusion ratio) and CPHR (coarse powder, high extrusion ratio) than in FPLR (fine powder, low extrusion ratio).

At higher magnifications a rod-like precipitate can be observed at the IMC particle edges in CPLR and CPHR and it appears throughout the IMC particles in FPLR. This morphology is typical of TiB [34].

4.1.3.2 Composite Interfacial Layer Thickness Measurements

Two distinct interfacial sublayers can be identified around the reinforcement particles. The reaction layers are a result of interdiffusion between the matrix and the reinforcement. About ten measurements were made around each IMC particle to determine the thickness of the reaction layers. Figure 4.14 shows the micrographs of extruded and HIP'ed samples. The HIP'ing conditions that the composites were exposed to resulted in more reactions among the composite constituents. Interfacial reaction layer 2 in composite CPLR grew
larger, see Figure 4.14(a). The reaction layers tended to inter-connect between different matrix envelopes around IMC particle. More reactions occurred between the constituents of composite FPLR, this made it difficult to discern the two reaction sub-layers that are observed in the as extruded composites. Less of the matrix can be seen on the micrograph of FPLR due to its more extensive reaction with the reinforcement.

<table>
<thead>
<tr>
<th>Reaction layer</th>
<th>C1 (as extruded)</th>
<th>C1 (extruded and HIP'ed)</th>
<th>C2 (as extruded)</th>
<th>C3 (as extruded)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total reaction layer (µm)</td>
<td>Mean 30.125</td>
<td>Median 38.1</td>
<td></td>
<td>Mean 29</td>
</tr>
</tbody>
</table>
Figure 4.14: SEM micrographs of extruded and HIP'ed samples, (a) CPLR, and (b) FPLR.
4.1.3.3 Composition analysis of the composites

Figure 4.15(a) is a micrograph of CPLR showing how spots were chosen within the composite microstructure for composition analysis using EDX; it also shows the extent of interfacial reactions. The interfacial reactions have led to semi-continuous porosity along some parts of the interface. The Al and Ti compositions were mapped along the major dimension within the IMC particle. Figures 4.15 (b) and (c) give X-ray plots of spots A and B marked on the micrograph.

The results of composition analysis of the three composites are shown in Figures 4.16, 4.17 and 4.18. The composition is expressed in terms of aluminum atomic percent, the remainder is titanium. The EDX spectra shown in Figures 4.15 (b) and (c) do not show any boron peaks because the sensitivity of EDX towards light elements is very limited, thus a determination of boron concentration was not feasible.

The Al content increases with distance from the interface, towards the IMC particle center. On the other hand, Al content decreases as the distance from the interface increases towards the matrix. The Al concentration in FPLR shows that there is more inter-diffusion between the reinforcement and the matrix. The Al content inside the particle is lower than that of the as-formulated near-gamma TiAl (46 at% Al) and more Al is found in the Ti matrix.

The extent of diffusion in this composite is favored by the small IMC particle size which results in higher surface area. The lower Al content inside the IMC particles is also manifested by the absence of the lamellar $\alpha_2 + \gamma$ morphology; this is shown in Figure 4.19.
Figure 4.15: (a) An SEM micrograph of composite CPLR showing how analysis spots were chosen for EDX analysis. (b) and (c) are EDX spectra of spots A and B marked on (a), showing the Al and Ti peaks.
Figure 4.16: Chemical composition analysis of as-extruded CPLR showing change in Al concentration.

Figure 4.17: Al concentration profile in FPLR obtained using EDX analysis.

Figure 4.18: Al concentration profile in CPHR (as-extruded) obtained using EDX analysis.
The Al concentration ranges for the particle edge, interfacial layer 1, interfacial layer 2 and the matrix are given in Table 4.5.

The Al content of the particle edges and reaction layer 1 largely corresponds to the Ti₃Al phase, in accordance with the Ti-Al phase diagram. An assessed phase diagram of the Ti-Al system is given in Fig. 20.

Table 4.5: Al content ranges for different composite areas.

<table>
<thead>
<tr>
<th>Composites</th>
<th>Aluminum concentration (at%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Particle edge</td>
</tr>
<tr>
<td>CPLR</td>
<td>16.06 - 30.45</td>
</tr>
<tr>
<td>FPLR</td>
<td>4.18 - 13.73</td>
</tr>
</tbody>
</table>
The Al content of reaction layer 2 (in CPLR and CPHR) corresponds mainly to a composition within the α-phase region. The data of Table 4.5 further illustrates the higher inter-diffusion between the components of FPLR; the matrix of this composite contains a higher concentration of Al. CPHR tends to have slightly higher Al concentration in reaction layer 2 and the matrix component compared to CPLR. The reaction sublayers in composite CPLR are marked in Figure 4.15(a). Area C is reaction sublayer 1, D is reaction sublayer 2 and E is the matrix component. Area F is an α-Ti region with Al concentration of about 3 at%, this region was not clearly defined throughout the composite microstructure in the specimens examined, this is evident from the particle shown at the right hand corner of the micrograph.

The composition profiles of extruded plus HIP'ed composites are shown in Figures 4.21 and 4.22.
Figure 4.21: Al content in extruded plus HIP'ed CPLR obtained with EDX.

Figure 4.22: EDX analysis of extruded plus HIP'ed FPLR.
The Al composition of extruded and HIP'ed CPLR dropped at the particle edges due to exposure to the HIP'ing conditions. The composition in other areas of the composite did not show significant change. Figure 4.21 shows a decrease in Al concentration within the IMC particles. The interfacial region expanded to cover most of the composite area outside the reinforcement. The matrix region covers a smaller area compared to the area it covers in its as-extruded counterpart (compare Figs. 4.11 and 4.14 (b)).

4.1.3.4 Composite geometrical considerations

The results obtained from particle size measurements are summarized in Table 4.6.

The measured reinforcement (IMC) volume fractions in the composites are lower than the nominal volume fractions. Composite FPLR exhibits the largest discrepancy between the nominal and measured IMC volume fractions. The low volume fraction is believed to be due to the dissolution of the IMC and the reaction between the IMC and the matrix. Composites CPLR and CPHR were formulated with the same IMC powder particle size distribution. The slightly higher IMC particle aspect ratio of CPHR compared to CPLR is due to the higher extrusion ratio used in the fabrication of CPHR (die ratios 19.8:1 for CPHR compared to 14.1:1 for CPLR). The other effect of the higher extrusion ratio used in the processing of CPHR appears to be a reduction in the porosity of composite CPHR; it exhibited slightly lower porosity within the reinforcing phase than CPLR.

Figure 4.23 illustrates how the particle radius used in the calculation of the edge-to-edge spacing was determined; by taking into account the thickness of the reaction sublayers.
Table 4.6: Particle size analysis of the composites.

<table>
<thead>
<tr>
<th>Composite</th>
<th>Volume fraction</th>
<th>$d_e$ ($\mu$m)</th>
<th>$d_{\text{major}}/d_{\text{min}}$</th>
<th>$\lambda$ ($\mu$m)</th>
<th>$\lambda-2r_1^*$ (\mu m)</th>
<th>$\lambda-2r_2^*$ (\mu m)</th>
<th>$\lambda-2r_3^*$ (\mu m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPLR mean</td>
<td>17.68</td>
<td>153.54</td>
<td>2.01</td>
<td>342.85</td>
<td>217.48</td>
<td>194.12</td>
<td>168.29</td>
</tr>
<tr>
<td>std. dev.</td>
<td>4.91</td>
<td>78.05</td>
<td>0.753</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FPLR mean</td>
<td>9.004</td>
<td>37.68</td>
<td>2.29</td>
<td>106.19</td>
<td>75.42</td>
<td>54.36</td>
<td>28.06</td>
</tr>
<tr>
<td>std. dev.</td>
<td>4.18</td>
<td>23.09</td>
<td>1.89</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CPFHR mean</td>
<td>14.93</td>
<td>156.40</td>
<td>2.12</td>
<td>385.84</td>
<td>258.14</td>
<td>233.01</td>
<td>200.98</td>
</tr>
<tr>
<td>std. dev.</td>
<td>2.91</td>
<td>64.34</td>
<td>1.73</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

de is the IMC equivalent diameter
$\lambda$ ($\mu$m) is IMC center-to-center spacing
$\lambda-2r_1^*$ is reinforcement edge-to-edge spacing based on the radius of IMC particle
$\lambda-2r_2^*$ is reinforcement edge-to-edge spacing based on the radius of IMC particle + thickness of reaction layer 1.
$\lambda-2r_3^*$ is reinforcement edge-to-edge spacing based on the radius of IMC particle + sum of thicknesses of reaction layers 1 and 2.

Figure 4.23: A schematic of an IMC particle showing the measurement of particle radius.
4.2 MECHANICAL PROPERTIES

Evaluation of the mechanical properties of the matrix and composites comprised of micro-hardness, compressive and tensile deformation.

4.2.1 MICROHARDNESS MEASUREMENTS

Microhardness results of both the as-extruded and the extruded plus HiP'ed matrix are given in Table 4.7.

<table>
<thead>
<tr>
<th>Vicker's hardness</th>
<th>As-extruded matrix</th>
<th>Extruded + HIP'ed matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>227.86</td>
<td>230.65</td>
</tr>
<tr>
<td>Median</td>
<td>229.5</td>
<td>232.6</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>17.93</td>
<td>24.7</td>
</tr>
</tbody>
</table>

The hardness of the matrix did not change with HiPing.

The results for as-extruded composites are given in Table 4.8. Vicker's microhardness results of extruded and subsequently HIP'ed composites CPLR and FPLR are given in Table 4.9.

Limited magnification of the sample during microhardness measurements made it impossible to differentiate between the interfacial sublayers of composite FPLR, thus the microhardness measurements of this composite covers the whole interfacial region. The higher hardness of the matrix component in FPLR is due to the higher concentration of aluminum in the matrix compared to the other composites.
### Table 4.8: Vicker's hardness data for as-extruded composites

<table>
<thead>
<tr>
<th>Composite</th>
<th>Composite area</th>
<th>Vicker’s Hardness</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>Median</td>
<td>Standard deviation</td>
<td></td>
</tr>
<tr>
<td>CPLR</td>
<td>matrix</td>
<td>306.82</td>
<td>309.59</td>
<td>35.712</td>
<td></td>
</tr>
<tr>
<td></td>
<td>IMC particle</td>
<td>613.828</td>
<td>600</td>
<td>62.265</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer 1</td>
<td>413.71</td>
<td>415.2</td>
<td>44.85</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer 2</td>
<td>319.07</td>
<td>329</td>
<td>22.97</td>
<td></td>
</tr>
<tr>
<td>FPLR</td>
<td>matrix</td>
<td>367.2</td>
<td>364.8</td>
<td>21.379</td>
<td></td>
</tr>
<tr>
<td></td>
<td>IMC particle</td>
<td>551.575</td>
<td>554.45</td>
<td>85.256</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer (1+2)</td>
<td>404.529</td>
<td>404.7</td>
<td>42.956</td>
<td></td>
</tr>
<tr>
<td>CPHR</td>
<td>matrix</td>
<td>246.6</td>
<td>243.4</td>
<td>15.74</td>
<td></td>
</tr>
<tr>
<td></td>
<td>IMC particle</td>
<td>556.27</td>
<td>564.7</td>
<td>63.601</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer 1</td>
<td>323.98</td>
<td>333.6</td>
<td>40.291</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer 2</td>
<td>287.23</td>
<td>279.65</td>
<td>25.32</td>
<td></td>
</tr>
</tbody>
</table>

### Table 4.9: Vicker's hardness data of extruded and HIP’ed composites

<table>
<thead>
<tr>
<th>Composite</th>
<th>Composite area</th>
<th>Vicker’s Hardness</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>Median</td>
<td>Standard deviation</td>
<td></td>
</tr>
<tr>
<td>CPLR</td>
<td>matrix</td>
<td>257.627</td>
<td>255.6</td>
<td>19.605</td>
<td></td>
</tr>
<tr>
<td></td>
<td>IMC particle</td>
<td>583.59</td>
<td>591.15</td>
<td>51.686</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer 1</td>
<td>423.52</td>
<td>413.55</td>
<td>45.857</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer 2</td>
<td>350.818</td>
<td>354.7</td>
<td>30.427</td>
<td></td>
</tr>
<tr>
<td>FPLR</td>
<td>matrix</td>
<td>368.118</td>
<td>379.2</td>
<td>29.658</td>
<td></td>
</tr>
<tr>
<td></td>
<td>IMC particle</td>
<td>865.156</td>
<td>840.6</td>
<td>148.724</td>
<td></td>
</tr>
<tr>
<td></td>
<td>reaction layer</td>
<td>465.98</td>
<td>466.65</td>
<td>44.989</td>
<td></td>
</tr>
</tbody>
</table>
4.2.2 Room Temperature Compression Test Results of IMC Reinforcement and the Composites

4.2.2.1 Room Temperature Compression Test Results of IMC

The consolidated specimens of each IMC (IMC1 and IMC2) that were tested in compression fractured before yielding. The measured fracture stress of IMC1 is 1024 MPa. The fracture strength of IMC2 is 1036 MPa. The testing of IMC2 was stopped after the specimen started cracking (before complete failure occurred).

4.2.2.2 Compression Test Results of the IMC-reinforced Metal Matrix Composites

All the metal matrix composites were tested in compression in order to determine their deformation behavior. Two specimens of each composite were tested. Engineering stress and strain data obtained from compression tests were used to calculate true stress and true strain for each material tested. The true strain, ε, may be determined from the engineering strain, e, by applying equation 4.1 to the measured data [28].

\[ \varepsilon = \ln(e + 1) \]  \hspace{1cm} (4.1)

The relationship between the true stress, σ, and the engineering stress, s, is given by the following equation:

\[ \sigma = s(e + 1) \]  \hspace{1cm} (4.2)

Examples of true compressive stress-strain curves of the composites are given in Figures 4.24 - 4.26.

Composite FPLR exhibited the highest strength followed by CPLR and then CPHR.
Figure 4.24: Stress-strain curve of a CPLR composite specimen tested in compression.

Figure 4.25: A stress-strain curve of FPLR tested in compression.
4.2.3 ANALYSIS OF DEFORMATION AND STRENGTH DATA

The flow curves of metals in the region of uniform plastic deformation can be expressed by the following power hardening relationship [34].

\[ \sigma = K\varepsilon^n \quad (4.3) \]

where \( n \) is the strain-hardening exponent and \( K \) is the strength coefficient. Equation (4.3) can be re-written as follows:

\[ \log \sigma = \log K + n \log \varepsilon \quad (4.4) \]

A plot of \( \sigma \) vs \( \varepsilon \) on log-log axes (or \( \log \sigma \) vs \( \log \varepsilon \) on linear axes) should give a straight line, with an intercept of \( \log K \) at \( \log \varepsilon = 0 \) and a slope \( n \). Hence, \( K \) is the true stress at
\( \varepsilon_p = 1 \). For plastic deformation, values of \( n \) typically fall within the range of 0.0 to 0.6. The flow curves of the matrix (tested in tension) and the composites were used to determine true stress as a function of plastic strain up to maximum load. Log stress vs log strain curves are given in Figures 4.27 - 4.31.
Figure 4.27: Log-stress - log-strain for the as-extruded matrix, (a) specimen A and (b) combined data of specimens A and B.
Figure 4.28: Log stress-log strain for extruded plus HIP'ed matrix.
Figure 4.29: Log-stress - log-strain for CPLR, (a) for specimen A and (b) combined data of specimens A and B.
Figure 4.30: A log stress vs log strain graph for FPLR, (a) specimen B and (b) combined data of specimens A and B.
Figure 4.31: Log stress-log strain curve for composite CPHR, (a) data for specimen A and (b) combined data (for A and B).
Table 4.10 is a summary of plastic deformation data of the matrix and the composites for compression tests.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>0.2% Yield Strength (MPa)</th>
<th>Strength Coefficient, K (MPa)</th>
<th>Strain Hardening Exponent, n</th>
<th>Coefficient of Variation, r²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix as extruded</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>specimen A</td>
<td>475.8</td>
<td>697.15</td>
<td>0.065</td>
<td>0.959</td>
</tr>
<tr>
<td>specimen B</td>
<td>521.7</td>
<td>809.16</td>
<td>0.076</td>
<td>0.947</td>
</tr>
<tr>
<td>Average of A and B</td>
<td>498.8</td>
<td>753.16</td>
<td>0.070</td>
<td></td>
</tr>
<tr>
<td>Combined data</td>
<td></td>
<td>764.3</td>
<td>0.073</td>
<td>0.662</td>
</tr>
<tr>
<td>Matrix (extruded + HIP'ed)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>409.8</td>
<td>681.6</td>
<td>0.085</td>
<td>0.972</td>
</tr>
<tr>
<td>CPLR specimen A</td>
<td>827.0</td>
<td>1791.8</td>
<td>0.140</td>
<td>0.911</td>
</tr>
<tr>
<td>specimen B</td>
<td>839.0</td>
<td>2151.7</td>
<td>0.169</td>
<td>0.948</td>
</tr>
<tr>
<td>Average of A and B</td>
<td>833.0</td>
<td>1971.8</td>
<td>0.154</td>
<td></td>
</tr>
<tr>
<td>Combined data</td>
<td></td>
<td>1950.6</td>
<td>0.150</td>
<td>0.844</td>
</tr>
<tr>
<td>FPLR specimen A</td>
<td>1013</td>
<td>2352.9</td>
<td>0.157</td>
<td>0.977</td>
</tr>
<tr>
<td>specimen B</td>
<td>1283.8</td>
<td>2683.8</td>
<td>0.145</td>
<td>0.962</td>
</tr>
<tr>
<td>Average of A and B</td>
<td>1148.4</td>
<td>2518.4</td>
<td>0.151</td>
<td></td>
</tr>
<tr>
<td>Combined data</td>
<td></td>
<td>2266</td>
<td>0.107</td>
<td>0.413</td>
</tr>
<tr>
<td>CPHR specimen A</td>
<td>741.3</td>
<td>1733.7</td>
<td>0.149</td>
<td>0.958</td>
</tr>
<tr>
<td>specimen B</td>
<td>795.1</td>
<td>1791.8</td>
<td>0.136</td>
<td>0.974</td>
</tr>
<tr>
<td>Average of A and B</td>
<td>768.2</td>
<td>1762.8</td>
<td>0.143</td>
<td></td>
</tr>
<tr>
<td>Combined data</td>
<td></td>
<td>1655.5</td>
<td>0.125</td>
<td>0.906</td>
</tr>
</tbody>
</table>

† The average squared deviation from the linear regression-determined mean; r = 1.0 means all points fall on the computed best-fit line.
The deformation behavior of all the composites and the matrix obey the flow stress-plastic strain power hardening relationship given by equation (4.3). The power law constants, K and n, for each specimen tested were given in Table 4.10. Individual constants were determined for each specimen; these values were averaged to give an average value for each material. Additionally, the flow stress and plastic strain data of different specimens (of each composite) were combined and the combined data was used to determine the power law constants. The correlation coefficient, r², from the linear regression analysis is also given. The correlation coefficients for the combined data are much lower than those of curves plotted using data from the individual specimens; with FPLR showing the largest deviation. The constants obtained using combined data do not accurately represent the individual specimens.

Composite FPLR exhibits the highest strength and strength coefficient. The strength of the base matrix is significantly increased by the presence of the reinforcing IMC phase and the presence of a significant concentration of Al which has diffused from the particles to the matrix. The high strength of composite FPLR is largely due to the small IMC particle spacing resulting from the fine IMC precursor. The strength values (0.2% yield strength and strength coefficient) of CPHR are lower than those of CPLR, the difference is believed to be due to the smaller inter-reinforcement spacings in the latter (Table 4.6).

A portion of the strengthening found in the composites is also believed to be derived from solid solution strengthening, which resulted from the diffusion of Al into the matrix. EDX analysis showed the presence of Al in the matrix phase, with significant concentration of aluminum found in the matrix of composite FPLR. The strengthening effect of solid solution is also supported by hardness data. Higher matrix hardness values were measured in the composites compared to unreinforced matrix. Despite exhibiting the highest strength, composite FPLR does not exhibit a high strain hardening coefficient.
compared to the other composites; instead, the strain hardening coefficients obtained for
the three composites are about the same. However, the IMC reinforcement improved the
strain hardening coefficient of the matrix by about two times.

4.2.4 ROOM TEMPERATURE TENSILE TEST RESULTS OF MATRIX AND
COMPOSITES

4.2.4.1 TENSILE DEFORMATION

Tensile tests were performed on the matrix and composites. Examples of stress versus
strain curves obtained in these tests are shown in Figures 4.32 and 4.33. The tensile
stress-strain curves of the composites and extruded and HIP'ed matrix are combined in
Figure 4.33.

![Stress-strain curve of the as-extruded matrix](image)

Figure 4.32. Stress - strain curve of the as-extruded matrix.
Figure 4.33: Stress-strain curve of composites and extruded and HIP'ed matrix (curves for CPLR, FPLR and CPHR specimens end at points of fracture).

Extruded plus HIP'ed matrix specimens exhibited a slightly higher ductility and lower strength than the as extruded specimen. The composites exhibited brittle stress-strain behavior; all the specimens fractured prior to yielding. Table 4.11 gives a summary of tensile data of the matrix and the three composites.

The matrix exhibited more ductility in the extruded plus HIP'ed condition and lower strength compared to the as extruded condition. Composites CPLR and FPLR were tested in the extruded plus HIP'ed condition whereas CPHR was tested in the as extruded condition only. Composite FPLR exhibited the highest elastic modulus and fracture strength; and CPHR exhibited the highest ductility and the lowest modulus of the three composites. (Note: The slope of FPLR appears smaller than that of CPLR in Fig. 4.33, this is due to testing machine misalignment during the initial portions of the test. The modulus values of the composites were calculated from the linear portions of the stress-strain curves, typically established after strains of about 0.001.) It is difficult to draw
conclusions about the strengthening involved in these composites since no yielding occurred when the composites were tested in tension.

Table 4.11: A summary of tensile data of matrix and composites

<table>
<thead>
<tr>
<th>Composite</th>
<th>Young's Modulus (GPa)</th>
<th>Fracture stress (MPa)</th>
<th>Fracture strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td>109.34</td>
<td>566</td>
<td>6.7</td>
</tr>
<tr>
<td>specimen A*</td>
<td>115.95</td>
<td>610.8</td>
<td>8.35</td>
</tr>
<tr>
<td>Average values</td>
<td>112.6</td>
<td>588.4</td>
<td>7.5</td>
</tr>
<tr>
<td>Matrix**</td>
<td>115.8</td>
<td>482</td>
<td>9.17</td>
</tr>
<tr>
<td>CPLR - specimen A**</td>
<td>128.10</td>
<td>373.5</td>
<td>0.268</td>
</tr>
<tr>
<td>CPLR - specimen B**</td>
<td>120.64</td>
<td>375.4</td>
<td>0.315</td>
</tr>
<tr>
<td>Average values</td>
<td>124.37</td>
<td>374.4</td>
<td>0.292</td>
</tr>
<tr>
<td>FPLR - specimen A**</td>
<td>131.8</td>
<td>464.3</td>
<td>0.36</td>
</tr>
<tr>
<td>FPLR - specimen B**</td>
<td>136.98</td>
<td>485.6</td>
<td>0.357</td>
</tr>
<tr>
<td>Average values</td>
<td>134.39</td>
<td>475</td>
<td>0.359</td>
</tr>
<tr>
<td>CPHR - specimen A*</td>
<td>111.06</td>
<td>400.4</td>
<td>0.395</td>
</tr>
<tr>
<td>CPHR - specimen B*</td>
<td>105.51</td>
<td>408</td>
<td>0.468</td>
</tr>
<tr>
<td>Average values</td>
<td>108.28</td>
<td>404.2</td>
<td>0.432</td>
</tr>
</tbody>
</table>

* as extruded condition
** extruded and HIP'ed condition

4.2.4.2 Fractography of Matrix and Composites

Fracture surfaces of all the specimen tested in tension were examined using SEM to determine the nature of fracture of the different specimens tested. Micrographs of the matrix and the composites are given in Figure 4.34-4.37.
Figure 4.34: Fracture surface of: (a) as-extruded matrix and (b) extruded and HIP'ed matrix.
Figure 4.35: Fracture surface of composite CPLR, (a) at low magnification and (b) at higher magnification.
Figure 4.36: An SEM micrograph of a fracture surface in FPLR, (a) at low magnification and (b) at high magnification.
Figure 4.37: SEM micrograph showing the nature of fracture in composite CPHR, (a) at low magnification and (b) at higher magnification.
The matrix exhibits ductile fracture, with the as-extruded specimen showing less ductility than the extruded plus HIP'ed specimen. The micrographs of the composites show a combination of ductile fracture in matrix and brittle fracture, characterized by flat facets in IMC particle areas. On the higher magnification micrographs of the composites cracks can be observed going around and through the reinforcing particles. Figure 4.35 (a) shows dark spots, these areas may have acted as crack initiation sites.
5. DISCUSSION

5.1 MICROSTRUCTURE

5.1.1 REINFORCEMENT (IMC)

5.1.1.1 GENERAL FEATURES

Reaction synthesis resulted in near-gamma TiAl matrix composites with a fine dispersion of TiB$_2$ particulates, exhibiting a mean diameter of about 0.7 $\mu$m. The TiB$_2$ particulate size is related to the degree of exothermicity through the adiabatic temperature of the reaction synthesis. Martin [35] demonstrated that the size of the TiB$_2$ formed during synthesis of IMC increases with increasing TiB$_2$ volume fractions for volume fractions below 50%. This is due to the fact that higher TiB$_2$ volume fractions yield higher adiabatic temperatures [35].

The TiB$_2$ particulates within the IMC tend to segregate to grain boundaries of the near-$\gamma$ TiAl matrix. Segregation during solidification is influenced by size and cooling rate; the small size of TiB$_2$ particles favors segregation. Also, since the IMC was reacted in a vacuum atmosphere, it cooled slowly after synthesis, this too promotes TiB$_2$ segregation to the matrix grain boundaries.

5.1.1.2 PRESENCE OF CARBIDES IN THE IMC

The near-$\gamma$ gamma TiAl intermetallic composites (Ti-46 at%Al + 20 vol%TiB$_2$) contained large carbide particles. These unwanted particles are believed to be due to contamination from the graphite crucible used during IMC synthesis. Carbide formation has been found to be favored conditions which lead to small TiB$_2$ [35]. Since light
elements such as carbon and boron are difficult to analyze with EDX, it was not possible to obtain an accurate compositional analysis of the carbide particles. Nonetheless, the carbide particles are believed to be aluminum and/or titanium carbides. Vicker's microhardness measurements on the presumed carbide particles yielded very high microhardness values, and the particles cracked extensively after indentation.

5.1.2 COMPOSITES

5.1.2.1 GENERAL MICROSTRUCTURAL FEATURES OF THE COMPOSITES

The co-extrusion of a ductile titanium matrix with a hard near-$\gamma$ TiAl IMC reinforcing phase was externally successful. However, the desired commensurably-deformed microstructure was not achieved. A large flow strength differential between the composite constituents is believed to have resulted in the lack of extrusion-derived deformation in the IMC reinforcing phase. Load transfer from the ductile CP titanium matrix to the IMC was not sufficient to result in the deformation of the latter, this is evidenced by the matrix "envelope" observed around IMC particles.

Even though no rod-like boride morphology was observed in the as synthesized IMC, it is observed within the IMC particles in the composites. The rod-like morphology is typical of TiB [33], and it was observed around the inner edges of the IMC particles in composites CPLR and CPHR, and throughout the IMC particle in FPLR. Since titanium monoboride is stable in $\alpha$- and $\alpha_2$-Ti alloys and not in the gamma or near-gamma compositions, it is believed that TiB$_2$ converts to TiB as the aluminum concentration decreases in these areas. The exact composition of the rod-like morphology could not be determined with the analysis tools used in this study due to insensitivity of EDX to light elements such as boron.
Some porosity is observed in all the composites; it is attributed primarily to the initial porous nature of the IMC, its resistance to deformation, and to inter-diffusion between the composite constituents (Kirkendall porosity). The diffusion-contributed porosity occurs mainly around the interfacial region and also within the matrix, particularly in extruded and HIP'ed composite FPLR.

Different reinforcement spacings within the composites were obtained by using different IMC particle size distributions and extrusion ratios. The IMC initial particle size appears to have a more pronounced effect in determining the IMC reinforcement spacing for the chosen extrusion ratios. The extrusion ratios of 14:1 and 19:1 used in the study do not result in significant change in the IMC spacing; this was to be expected given the predicted spacings based on geometrical considerations. As shown in Figure 5.1, spacings become increasingly insensitive to extrusion ratio at values of 10 and greater.

![Graph showing the dependence of in-situ formed IMC spacing on extrusion ratio.](image)

Figure 5.1: Dependence of in-situ formed IMC spacing on extrusion ratio [36].
5.1.2.2 Composition Changes within the Microstructure

Energy dispersive X-ray microanalyses show that the highest concentration of Al is found around the center of each IMC particle, and it drops with increasing distance from the IMC particle center. The Al content within the IMC particles in composite FPLR is well below that of a near-gamma TiAl alloy, the maximum Al content within the IMC particles in this composite corresponds to compositions characteristic of single-phase Ti₃Al. The small IMC powder size and hence large surface areas increases the extent of diffusion of the composite components. This point suggests that processing changed the IMC microstructure in this composite, as evidenced by the absence of the lamellar morphology within the IMC particles of composite FPLR. Some Al was detected in the titanium matrix in all the composites, but composite FPLR shows higher concentrations Al solid solution in titanium. The small particle size and hence higher surface area resulted in higher diffusion rates in this composite.

5.1.2.3 Interfacial Reactions

Large reaction zones were observed in the composites of the present study, they can be attributed primarily to the high reactivity of the titanium matrix. Similar composites in which the Ti-6Al-4V alloy was used as a matrix showed very limited interfacial reaction between the matrix and the IMC reinforcement. Figure 5.2 is a micrograph of a Ti-6Al-4V + 20 vol%(Ti-46 at%Al + 30 vol% TiB₂) [35].

Other studies in which pure titanium and alloyed matrices were used with the same reinforcements also showed thicker reaction zones in composites with pure titanium matrices compared to those with alloyed matrices [22, 37].
It is evident from the extruded plus HIP'ed composites that exposure at high temperature increases inter-diffusion between the composite constituents. After HIP'ing at 1260°C for 4 hours; the thickness of reaction layer 2 in composite CPLR increased even though no significant change in the Al concentration within the IMC was detected. Except for a slight decrease of Al content on the particle edge, no significant change in the Al content in other areas of the composite microstructure was detected. Thickness of the reaction zone 1 (Ti₃Al) in CPLR did not grow after exposure at high temperature, this is probably due to the fact that there could be no further diffusion of either Ti or Al into or out of this phase. On the other hand, exposure at high temperature had a more pronounced effect on composite FPLR; the inter-diffusion between components increased, as illustrated by the decrease in Al content within the IMC particles of this composite and an increase in the thickness of the second reaction layer (solid solution α-Ti) which
covered a much larger matrix area around the reinforcing phase. There was not sufficient data to fit the interfacial growth kinetics of these composites to a model. Nonetheless, it is evident that the growth of the interfacial zone (α-Ti) tends to increase with increasing exposure to the elevated temperatures of processing.

5.2 MECHANICAL PROPERTIES

5.2.1 STRENGTH AND STRAIN HARDENING

The strengthening observed in these composites is believed to derived from two main sources, namely, (i) microstructural changes within the matrix due to the presence of the IMC reinforcement, and (ii) solid solution strengthening due to the diffusion of Al into the matrix phase. The former source of strengthening should exhibit a strong correlation to the inverse square root of the edge-to-edge interparticle spacing. Both sources of strengthening are discussed below.

5.2.1.1 EFFECT OF SPACING ON COMPOSITE STRENGTH

The strengthening due to coarse particles within metallic matrices is similar to that of two-phase alloys in that the two phases forming the microstructure deform differently, creating inhomogeneous plastic flow [31]. Inhomogeneous plastic flow results in the formation of deformation gradients which can be accommodated by introducing geometrically necessary dislocations. These "particle-stabilized" dislocations represent obstacles to flow and result in an increase in the yield and flow behavior of the alloy. The stress which must be applied to advance dislocations through these particle-stabilized dislocations is estimated to be [29]:

88
\[ \sigma_s = \sqrt{\frac{G_m E_p b}{\lambda_e}} \]  

(5.1)

where \( G_m \) = shear modulus of matrix

\( b \) = matrix Burger's vector

\( E_p \) = elastic modulus of the reinforcement

\( \lambda_e \) = edge-to-edge spacing

Equation (5.1) represents an estimate of the increment of strength due to the strengthening effect of the reinforcing particle; it predicts the dependence of composite flow stress on the inverse square root of the interparticle spacing. Equation (5.1) was used to estimate the magnitude of strengthening due to the particles, the results are given in Table 5.1. The value of \( E_p \) used in the calculations is 128.3 GPa, from section 5.2.2; the value of \( G_m = 46 \) GPa from reference [39] and the matrix Burgers vector = 0.295 nm from reference [39].

<table>
<thead>
<tr>
<th>Composite</th>
<th>( \lambda (\mu m) )</th>
<th>( \Delta \sigma_p ) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPLR</td>
<td>( \lambda_1 ) 217.48</td>
<td>89.47</td>
</tr>
<tr>
<td></td>
<td>( \lambda_2 ) 194.12</td>
<td>94.7</td>
</tr>
<tr>
<td></td>
<td>( \lambda_3 ) 168.29</td>
<td>101.71</td>
</tr>
<tr>
<td>FPLR</td>
<td>( \lambda_1 ) 75.42</td>
<td>151.94</td>
</tr>
<tr>
<td></td>
<td>( \lambda_2 ) 54.36</td>
<td>179</td>
</tr>
<tr>
<td></td>
<td>( \lambda_3 ) 28.06</td>
<td>249.1</td>
</tr>
<tr>
<td>CPHR</td>
<td>( \lambda_1 ) 258.14</td>
<td>82.1</td>
</tr>
<tr>
<td></td>
<td>( \lambda_2 ) 233.01</td>
<td>86.4</td>
</tr>
<tr>
<td></td>
<td>( \lambda_3 ) 200.98</td>
<td>93.07</td>
</tr>
</tbody>
</table>

\( \lambda_1 \) represents spacing due to radius of IMC particle

\( \lambda_2 \) represents spacing due to radius of reaction layer 1 + radius of IMC particle

\( \lambda_3 \) represents spacing due to radius of IMC particle + radius of reaction layers 1+2
The incremental strengths due to the particles increase with decreasing square root of the interparticle spacing.

5.2.1.2 SOLID SOLUTION STRENGTHENING

When solute atoms are introduced into a solvent, an alloy results which is stronger than the pure metal. Solute additions tend to raise the yield stress and the level of the stress-strain curve [28].

When metals dissolve in Ti, strong local and directional electronic bonds form between the solute and Ti atoms. Moving dislocations experience a strong pinning force when in the environment of the solute atoms [38]. Solute atoms can interact with dislocations through long-range interaction, modulus interaction, elastic interaction due to size differences between the solute and the solvent, short-range order interaction, and stacking-fault interaction. Solid-solution strengthening results from the resistance to dislocation motion through one or more of the mechanisms given above [28]. Figure 5.3 shows the effect of solid-solution strengthening of Ti [38].

![Figure 5.3: Solid-solution strengthening of titanium with binary and ternary additions of Al and Ga [38].](image-url)
Solid solution strengthening is difficult to quantify because it can result from several dislocation-solute atom interactions. Modulus and size effects produce an elastic dislocation-solute atom interaction energy [40]. Few models exist for quantifying solid-solution strengthening; a model by Fleischer [40], quantifies the size and modulus effects. A solute parameter, $\varepsilon_b$, which measures the relative size difference between the solute and solvent atoms, is defined as

$$\varepsilon_b = \frac{1}{a} \frac{\Delta a}{\Delta c}$$  \hspace{1cm} (5.2)

where $\Delta a$ is the change in lattice parameter due to addition of $\Delta c$ solute and $a$ is the solvent lattice parameter. A solute modulus parameter, $\varepsilon_G$, which takes into account differences in interaction due to screw and edge dislocations, is given by

$$\varepsilon_G = \frac{1}{G_A} \frac{\Delta G}{\Delta c}$$  \hspace{1cm} (5.3)

Also,

$$\varepsilon'_G = \frac{\varepsilon_G}{1 + 0.5|\varepsilon_G|}$$  \hspace{1cm} (5.4)

$G_A$ and $G_B$ are the shear moduli of the solvent and the solute, respectively. A solid-solution strengthening parameter, $\varepsilon_s$ is defined as

$$\varepsilon_s = |\varepsilon'_G - \beta \varepsilon_b|$$  \hspace{1cm} (5.5)

where $\varepsilon'_G$ takes into account differences in interaction due to screw and edge dislocations, $\beta$ is a constant and is approximately equal to 3. Thus the strengthening due to the solute can be represented by the following relation
\[ \Delta \sigma_m = \frac{2G_s \gamma_s \sqrt{c}}{700} \]  

(5.6)

where \( c \) is the concentration of solute in atomic fraction.

Aluminum concentrations ranging from about 0.4 to 4.7 in CPLR, 5 to 9.8 in FPLR and 1.8 to 3.9 at% in CPHR were measured within the composites using EDX. These concentrations were used to estimate the strengthening due to solid solution using the equations given above. The results are given in Table 5.2.

<table>
<thead>
<tr>
<th>Composite</th>
<th>( \Delta \sigma_m ) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPLR</td>
<td>28.95</td>
</tr>
<tr>
<td>FPLR</td>
<td>73.54</td>
</tr>
<tr>
<td>CPHR</td>
<td>26.36</td>
</tr>
</tbody>
</table>

Composite FPLR had the highest concentration of Al in Ti hence this composite has the highest estimated value of \( \Delta \sigma_m \).

### 5.2.1.3 STRAIN HARDENING

When a particle-reinforced composite is deformed, flow is interrupted by the reinforcing particles therefore the matrix shows increased work-hardening. The work-hardening of a material can be represented by the following equation

\[ \sigma_{flow} = \sigma_0 + \alpha G_m b \sqrt{\rho} \]  

(5.7)
where $\sigma_0$ is the flow stress of a material containing only statistical dislocation, $\alpha$ is a constant with values between 0.1 and 1.5 , $\rho$ is the density of geometrically necessary dislocations due to the plastically non-homogeneous nature of the microstructure.

Increasing plastic strain increases the dislocation density around the reinforcement in a local manner, at higher strains the dislocation density extends into the matrix [5]. The relation between plastic strain and dislocation density is given by

$$\rho \approx \frac{\varepsilon_p}{d}$$

(5.8)

where $\varepsilon_p$ is the plastic strain and $d$ is a characteristic dimension of the plastically inhomogeneous microstructure. In the case of particle-containing microstructures the edge-to-edge interparticle spacing is the characteristic dimension [29]. Equations (5.1) (5.7) and (5.8) can be combined to give the dependence of flow stress on the inverse square root of the interparticle spacing.

$$\sigma_{\text{flow}} = \sigma_0 + \alpha G_m b \sqrt{\frac{\varepsilon_p}{(\lambda - 2r^*)}}$$

(5.9)

where $\sigma_0$ is the flow stress at zero plastic strain. Equation (5.9) gives the particle-influenced strain-hardening increment of the composite flow stress. The second term of (5.9) is due to work-hardening, it is related to the strength coefficient, $K$, and the strain hardening coefficient $a$.

No significant difference was observed in the strain-hardening coefficients of the composites, nonetheless, the strain-hardening coefficient of the base matrix was significantly improved through compositing.
5.2.1.4 ADDITIVITY OF STRENGTHENING

The strengthening increments calculated above were added to the 0.2% flow strength of the base matrix and the sum was compared to the measured 0.2% flow strengths of the composites, \( \sigma_{\text{actual}} \). The results are summarized in Table 5.3.

Table 5.3: Summary of the strengthening data

<table>
<thead>
<tr>
<th>Composite</th>
<th>( \sigma_0 ) (base) (MPa)</th>
<th>( \Delta \sigma_{\text{SS}} ) (MPa)</th>
<th>( \lambda_1 )</th>
<th>( \lambda_2 )</th>
<th>( \lambda_3 )</th>
<th>( \Sigma \Delta \sigma^* ) (MPa)</th>
<th>( \sigma_{\text{actual}} ) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPLR</td>
<td>498.9</td>
<td>28.95</td>
<td>89.47</td>
<td>94.7</td>
<td>101.71</td>
<td>629.6</td>
<td>833</td>
</tr>
<tr>
<td>FPLR</td>
<td>498.9</td>
<td>73.54</td>
<td>151.94</td>
<td>179</td>
<td>249.1</td>
<td>821.54</td>
<td>1148.4</td>
</tr>
<tr>
<td>CPFR</td>
<td>498.9</td>
<td>26.36</td>
<td>82.1</td>
<td>86.4</td>
<td>93.07</td>
<td>618.3</td>
<td>768.2</td>
</tr>
</tbody>
</table>

\( \Sigma \Delta \sigma^* = \sigma_0 + \Delta \sigma_{\text{SS}} + \Delta \sigma_{p,\lambda 3} \)

The value of \( \Delta \sigma_p \) used in \( \Sigma \sigma \) is based on the interparticle spacing that takes into account the whole interfacial zone around the IMC particle. The value of the matrix flow strength used in Table 5.3 is that of the as-extruded matrix. The analysis given in Table 5.3 shows qualitative agreement between the predicted and actual values of strength. The difference between the measured strength and the summation of the strength contributions is believed to be due to interstitial solid solution strengthening and other microstructural features such as the matrix grain size. The presence of interstitial elements such as oxygen, nitrogen and carbon has a significant effect on the strength and ductility of commercially pure titanium [41, 42]. These interstitials increase the strength of Ti while lowering its ductility. The oxygen and nitrogen contamination within the composites is believed to be due to the powder-based approach used to make the composites. A concentration of 0.1% O equivalent (%O equiv. = %O + 2.0(%N) + 0.67(%C)) of interstitial elements in pure titanium increases the strength of pure Ti by about 120 MPa [42]. Strengthening due to matrix grain size could not be determined because it was
difficult to obtain grain size from the micrographs of the as-extruded matrix. Composite FPLR exhibited the highest value of $\Delta\sigma_m$ due to the high Al concentration in the Ti matrix of this composite. Also, this composite is expected to have the highest contribution of interstitial strengthening because of the much finer precursor powder size used in its fabrication.

5.2.2 PREDICTION OF ELASTIC MODULI AND COMPARISON WITH MEASURED DATA

The reinforcement of commercially pure titanium with high stiffness IMC improves the stiffness of the titanium matrix, as anticipated. The elastic moduli of the IMC and the composites can be predicted from the Hashin-Shtrikman model [43]. This model can be used to predict the modulus of a two-phase system, with a continuous phase, the matrix, and a discontinuous phase, the reinforcement. The model gives lower and upper bounds of the elastic modulus, based on the shear and bulk moduli according to the following equations:

$$K_u = K_1 + \frac{V_2}{\frac{1}{K_2 - K_1} + \frac{3(1 - V_2)}{3K_1 + 4G_1}} \quad (5.10)$$

$$K_u = K_2 + \frac{1 - V_2}{\frac{1}{(K_1 - K_2)} + \frac{3V_2}{3K_2 + 4G_2}} \quad (5.11)$$

$$G_L = G_1 + \frac{V_2}{\frac{1}{(G_2 - G_1)} + \frac{6(K_1 + 2G_1)(1 - V_2)}{5G_1(3K_1 + 4G_1)}} \quad (5.12)$$
\[ G_U = G_2 + \frac{1 - V_2}{(G_1 - G_2) + \frac{6(K_2 + 2G_2)V_2}{5G_2(3K_2 + 4G_2)}} \]  
\[ (5.13) \]

In the relations given above, \( K_L \) and \( G_L \) are the lower bounds of the bulk and shear moduli, \( K_U \) and \( G_U \) are the upper bounds of the bulk and shear moduli, and \( V \) is the volume fraction. The subscripts 1 and 2 refer to the continuous phase (matrix) and discontinuous phase (reinforcement) respectively. Upper and lower bounds on the elastic modulus can be calculated from the bounds for bulk and shear moduli using the relations

\[ E = \frac{9KG}{3K + G} \]  
\[ (5.14) \]

\[ K = \frac{E}{3(1 - 2v)} \]  
\[ (5.15) \]

\[ G = \frac{E}{2(1 + v)} \]  
\[ (5.16) \]

where \( v \) Poisson's ratio. Constants used to predict the moduli of the composites and the reinforcement in the present study are given in Table 5.4.

<table>
<thead>
<tr>
<th>Component</th>
<th>E(GPa)</th>
<th>G(GPa)</th>
<th>K(GPa)</th>
<th>( v )</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiB₂</td>
<td>538(^1)</td>
<td>240</td>
<td>236</td>
<td>0.12</td>
</tr>
<tr>
<td>TiAl</td>
<td>162(^1)</td>
<td>65</td>
<td>108</td>
<td>0.25</td>
</tr>
<tr>
<td>CP. Ti</td>
<td>109(^2)</td>
<td>41</td>
<td>107</td>
<td>0.33(^3)</td>
</tr>
</tbody>
</table>

\(^1\) from reference [29]  
\(^2\) measured  
\(^3\) from reference [44]
The values of the moduli given in Table 5.4 were used to calculate the shear and bulk moduli of 20 vol%TiB₂ in TiAl (using equations 5.15 and 5.16). $E_L$ and $E_U$ for the IMC were predicted to be 201.5 GPa and 215.4 GPa, respectively. These values give an average elastic modulus of 208 GPa.

The predicted lower and upper bounds of the shear and bulk modulus of the IMC were used together with the $K$ and $G$ values of commercially pure titanium given in Table 5.4 to predict the elastic modulus of the CP titanium + IMC composites. Lower and upper bounds of the elastic were predicted to be 123.3 GPa and 133.2 GPa, respectively, with the mean of these values being 128.3 GPa. The elastic moduli as calculated from the stress-strain curves of the three composites (CPLR, FPLR and CPHR) ranged from 105 to 134 GPa, in reasonable agreement with the Hashin-Shtrikman model's predictions. The three composites should have the same elastic modulus value because they were all formulated with the same nominal volume fraction of IMC (0.20). The differences are probably due to microstructural changes that are due to partial dissolution of the IMC that occurred during processing.

5.2.3 Fracture Behavior

All the composites exhibited brittle behavior in tension. The reduced ductility of the composites relative to the unreinforced matrix material can be attributed to the following factors:

1. An inevitable reduction due to the activation of new sources of strengthening, both intended (e.g., composite strengthening due to the presence of the brittle IMC) and unintended (e.g. due to solid solution strengthening from dissolved aluminum and oxygen).

2. Due to processing-based flaws present in the microstructure such as pores and cracks.
Unfortunately, the inherent ductility and toughness of a composite can be shadowed by the latter contribution such that extremely low "apparent" values are obtained, as manifested by, for example, fracture prior to yielding. This difficulty is partially alleviated by testing in compression, where the applied stresses tend to press together opposite sides of flaws such as cracks and pores. Thus yielding can be observed when "extrinsically" brittle materials are tested in compression.

Much higher ductilities were obtained when the composites were tested in compression, and fracture occurred at much higher stresses. All the cylindrical specimens tested exhibited compressive failure at about 45 degrees to the specimen axis, whereas the fracture plane in tension was oriented normal to the applied tension stress, typical of brittle behavior [34].

Even though the composites exhibited brittle behavior macroscopically, fractography showed ductile failure on a microscopic scale. The matrix exhibited ductile failure whereas the reinforcement areas showed brittle failure.

No tests were performed to determine the bond strength of the interfaces in the different composites; however, the thick reaction layers measured in the composites combined with the voids observed around the interfacial region, would be expected to lower the composites' resistance to fracture. Studies done by Reeves et al. on particulate SiC-reinforced CP titanium composites showed decreasing fracture toughness with increasing reaction layer thickness [45].

Micrographs of fracture surfaces taken after tensile deformation of the composites show that most of the cracking occurs around the reinforcing particle, at the reinforcement/matrix interface. Some of the cracks seem to originate from within the reinforcing IMC particles. The interface is further weakened by the presence of continuous porosity around some of the IMC particles. In some cases it may not be
thickness of the reaction layer as such that causes failure but the ease of formation of the crack, especially if the cracks first form parallel to the reaction layer [45]. This effect would be expected to be more pronounced in composites CPLR and CPHR because they contained larger continuous pores around the interfacial region. Poor interfacial bonding lowers the elastic modulus and work-hardening rate because of reduced load transfer between the composite constituents.
6. CONCLUSIONS

1. The co-extrusion of CP Ti with 20 vol% of a hard near-gamma TiAl IMC reinforcing phase was successful. The flow stresses of the ductile matrix and the hard IMC reinforcement were not adequately matched during high temperature extrusion; as a result, the desired commensurably-deformed microstructure was not obtained.

2. The Ti + IMC composites were characterized by extensive interfacial reactions, particularly, in the composite formulated with fine IMC precursor powders.

3. The strengthening is due mainly to interactions between the reinforcement and dislocations during deformation. The magnitude of this incremental strength correlates with the inverse square root of the IMC interparticle spacing. Solid solution strengthening, due to the diffusion of Al from the reinforcement into the matrix, was found to be an additional contribution to strength in all the composites examined.
REFERENCES


VITA

The author, Thobeka Portia Pete, was born on May 21, 1962 at Vrede, Orange Free State, South Africa. She graduated from the University of Fort Hare, South Africa, in 1986 with a Bachelor of Science degree in Physics and Chemistry and a Diploma in Higher Education (mathematics and science option). After graduating from Fort Hare she worked for the South African Committee for Higher Education where she wrote physical science workbooks for high school seniors. In January of 1988 she joined the Argus Company in Johannesburg, where she was a printing technologist up to the end of June 1989. She was awarded a scholarship by the Institute of International Education to study Metallurgical Engineering at the University of Kentucky. In August of 1992 she graduated with a bachelor's degree in Materials Engineering from the University of Kentucky. In the fall of 1992 she joined the department of Materials Science and Engineering at Virginia Polytechnic Institute and State University as a graduate student. She received a Master’s degree in Materials Science and Engineering from V.P.I & S.U. in December 1994.