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MICROSTRUCTURAL EVOLUTION IN LASER AND ELECTRON BEAM WELDS ON SiC-REINFORCED A356 ALUMINUM

DISSERTATION

Presented in Partial Fulfillment of the Requirements for
the Degree of Doctor of Philosophy in the Graduate
School of The Ohio State University

By
Thomas J. Lienert, B.S., M.S.

The Ohio State University
1998

Dissertation Committee:

Professor Hamish L. Fraser, Adviser
Professor Carroll E. Mobley
Professor Charles H. Drummond, III
Professor Jerald R. Brevick

Approved by
Adviser
Department of Materials Science and Engineering
ABSTRACT

A comparative study of laser beam welding (LBW) and electron beam welding (EBW) of cast A356/SiC/15p aluminum metal matrix composite (Al-MMC) has been completed. Laser and electron beam welds have been made at identical powers, rapid travel speeds (up to 200 ipm) and focusing conditions to allow direct comparison of the effects of the welding process characteristics with the resulting microstructures. Microstructures of the welds as well as the base material were characterized using optical microscopy (OM) with image analysis, scanning electron microscopy (SEM) with X-ray energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), and transmission electron microscopy (TEM) with EDS with the goal of understanding the microstructural evolution of the welds. Microstructural evidence suggests that the SiC particles dissolve into a homogeneous solution at high temperatures during LBW, and Al₄C₃ precipitates from the solution during cooling. Conversely, for EBW, dissolution and displacement of the SiC is limited, and relatively little Al₄C₃ is formed. Variations in microstructures of the EB and LB welds made using identical powers, travel speeds and focusing conditions are attributed to the differences in the thermal cycles experienced with the two welding processes. In turn, the disparities in thermal cycles are rationalized in terms of: a) the differences in the physics of beam/substrate energy transfer for the two processes and b) pressure-temperature relations (EBW is performed in vacuum while LBW is performed near...
atmospheric pressure). Results of the microstructural analysis are combined with arguments based on physical and thermodynamic properties of the Al alloy and SiC to develop qualitative models for the microstructural evolution in the EB welds and the different regions of the LB welds on the Al-MMC.
DEDICATION

To Kellie
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VITA

November 30, 1958.................................................. Born - Buffalo, NY.

1981................................................................. A.A.S. Degree in Materials Science
Technology, Erie Community College.
Buffalo, NY.

1981 - 1989........................................................ Welding Research Technician
Sandia National Laboratories
Albuquerque, NM.

1989 - 1992........................................................ Senior Research Technician
Edison Welding Institute
Columbus, OH.

1992................................................................. B.S. Degree in Welding Engineering.
The Ohio State University
Columbus, OH.

1995................................................................. M.S. Degree in Materials Science and
Engineering, The Ohio State University
Columbus, OH.

1997 - 1998........................................................ Research Engineer
Edison Welding Institute
Columbus, OH.

PUBLICATIONS

1. T.J. Lienert, J.M.K. Wiezorek, H.L. Fraser and W.A. Baeslack III, “Microstructures and
Mechanical Properties of Inertia-Friction W elds on 8009/SiC/11p,” Joining of Advanced

2. T.J. Lienert, J.M.K. Wiezorek, H.L. Fraser and W.A. Baeslack III. “TEM
Characterization of Microstructures in Laser Beam Welds on A356/SiC/15p.” Joining


FIELDS OF STUDY

Major Field: Materials Science and Engineering

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                                                          Prof. G. Meyrick

Studies in Chemical Metallurgy............................... Prof. K. Sandhage
                                                          Prof. R. Rapp

Studies in Mechanical Metallurgy............................. Prof. G. Duehn
                                                          Prof. G. Meyrick

Studies in Electron Microscopy............................... Prof. H. Fraser
                                                          Prof. W. Clark

Studies in Crystallography, Crystal Physics
and Crystal Chemistry........................................... Prof. S. Dregia

Studies in Solidification...................................... Prof. C. Mobley

Studies in Welding Engineering............................... Prof. W. Baeslack III
                                                          Prof. C. Albright
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CHAPTER 1

INTRODUCTION

One of the major goals of Materials Science is to develop and utilize relationships between the composition and processing of a material and the resulting microstructures and properties. Specifically, composition and processing can be manipulated to produce materials with desirable microstructures which allow favorable properties and performance. Modern advances in processing now allow creation of high performance materials with engineered microstructures and properties. Aluminum metal matrix composites (Al-MMCs) comprise one group such materials that offer increased stiffness relative to conventional Al alloys.

The effective use of Al-MMCs will often depend upon the ability to join them to similar and dissimilar materials. Since the properties of Al-MMCs derive from their unique microstructures, welding processes must be chosen that retain the microstructure by limiting grain growth, reinforcement clustering and chemical reactions between the reinforcement and the matrix. One potential approach to joining these materials involves the use of high-energy density welding processes such as laser beam welding (LBW) and electron beam welding (EBW) that allow for very rapid thermal cycles. The objectives of this work are to produce and characterize LB and EB welds on SiC-reinforced Al-Si alloys with a view toward developing an understanding of microstructural evolution in the welds. A second goal of this work is to determine mechanical properties of the welds.
Chapter 2 of this dissertation provides a review of several issues pertinent to welding of aluminum metal-matrix composites (Al-MMCs). The first section of the review provides an introduction to the production, properties and uses of Al-MMCs. In subsequent sections, phase equilibria in the Al-Si-C and the Al-Mg-Si systems are reviewed along with the crystallography of the phases found in these systems. In the final section, issues of microstructural development in cast Al-MMCs are discussed. Specifically, three major topics are outlined: (1) the thermodynamics of reactions between liquid Al alloys and SiC; (2) reinforcement redistribution during solidification of Al-MMCs; and (3) grain size vs. cooling rates for Al-MMCs. In Chapter 3, the information developed in the Chapter 2 is applied to welding of Al-MMCs. A review of papers concerning welding of Al-MMCs is presented.

Chapter 4 outlines the objectives of this work, while Chapter 5 provides details of the experimental work including the welding and characterization phases. Microstructures of the base material and the welds are characterized using optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM). Attempts are made to gauge mechanical properties via room temperature tensile testing of welds made on cast plates of SiC-reinforced Al-MMCs.

Results of the microstructural characterization are presented in Chapter 6. Microstructures of the EB and LB welds are found to differ dramatically despite the similarities in the processes. For example, the LB welds contain a large volume fraction of an undesirable phase, while the EB welds do not. Possible explanations for the differences in microstructures between the two processes are discussed in Chapters 7 and 8. The differences in microstructures are rationalized in terms of the fundamental differences in the physics of beam/substrate interactions of the two processes as well as the differences in peak temperatures of
the two processes due to pressure differences (EBW is performed in partial vacuum, while LBW is performed at atmospheric pressure).

Qualitative models for microstructural development of the different welds are proposed in Chapter 9. Microstructural evolution is discussed in terms of estimated time-temperature curves for the different regions and welding processes. Subsequently, the Al-Si-C ternary phase diagram is employed to develop models for microstructural evolution. The models are compared with the microstructural information found in Chapter 6 and with the results of mass balance calculations. Very good agreement is found between the proposed models and the results of characterization providing some assurance of the accuracy of the models.

An outline of the results of the experiments to produce casting for mechanical testing of LB and EB welds is given in Chapter 10. Attempts to measure tensile properties of the welds were not successful due to porosity and reinforcement clustering in the cast plates. Finally, conclusions are summarized in Chapter 11. EBW has proven capable of producing high-quality welds on SiC-reinforced Al-Si alloys. Appendices include a review of welding processes used to join Al-MMCs and details of the mass balance calculations.
CHAPTER 2

LITERATURE REVIEW

In this chapter, several issues pertinent to welding of aluminum metal-matrix composites (Al-MMCs) are reviewed. The first section of the review provides an introduction to the production, properties and uses of Al-MMCs. In subsequent sections, phase equilibria in the Al-Si-C and the Al-Mg-Si systems are reviewed along with the crystallography of the phases found in these systems. In the final section, issues of microstructural development in cast Al-MMCs are discussed. Specifically, three major topics are outlined: (1) the thermodynamics of reactions between liquid Al alloys and SiC; (2) reinforcement redistribution during solidification of Al-MMCs; and (3) grain size vs. cooling rates for Al-MMCs. A review of papers concerned directly with joining of Al-MMCs is provided in the next chapter.

2.1 Introduction to Al-MMCs

2.1.1 Nomenclature for Al-MMCs

Aluminum metal-matrix composites (Al-MMCs) are engineered materials that combine the properties of a ductile aluminum alloy matrix with those of a suitable reinforcement, usually a stiff ceramic. Both wrought and cast precipitation-hardenable aluminum alloys are used as the matrix, as well as the newer rapidly solidified (RS) alloys. Typical
reinforcement materials include boron (B), graphite (C), silicon carbide (SiC), and aluminum oxide (Al₂O₃). The reinforcement may take the form of chopped fibers (designated by subscript "c"), continuous fibers ("f"), whiskers ("w"), or particles ("p"). While much of the early research on Al-MMCs concentrated on continuous fiber materials, many recent investigations have focused on discontinuously reinforced (i.e. particle or whisker) aluminum (DRA) MMCs [2.1-2.4]. The growing interest in DRA-MMCs is primarily due to their greater ease and lower cost of production as well as their relatively isotropic properties.

Al-MMCs are normally specified according to a five part nomenclature developed by the Aluminum Association (AA) and later adopted by the American National Standards Institute (ANSI) [2.5]. In this specification, the matrix alloy is listed first followed sequentially by the reinforcement material, and the volume percent and shape of reinforcement, each separated by a slash. The heat-treatment designation, where appropriate, is placed after the percent and shape of reinforcement. For example, A356/SiC/15p-T6 refers to an A356 Al-casting alloy matrix containing 15 volume percent SiC particles that has been heat-treated to the -T6 condition.

2.1.2 Production Methods

The methods of production for Al-MMCs can be broadly divided into three categories according to the physical state of the matrix during incorporation of the reinforcement: solid phase processes, liquid phase processes, and two phase (solid/liquid) processes. Solid phase processes include powder metallurgy (P/M) methods, electrodeposition of Al alloys around reinforcement preforms, and diffusion bonding of alternate layers of fibers and Al foils [2.1.2.6]. The liquid phase processes can involve one of several casting or infiltration methods [2.7-2.10]. Rheocasting and spray codeposition methods are the most common of
the two phase processes [2.11.2.12]. Secondary processing in the form of rolling, extrusion, and forging of the DRA types of Al-MMCs is accomplished with only minor modifications to standard practices for Al alloys.

2.1.3 Engineering Properties

Al-MMCs provide unique combinations of properties not available with other materials. Additionally, the physical and mechanical properties of the composite can be tailored for different applications by varying the size, shape, and volume fraction of reinforcement [2.1]. Several physical and mechanical properties of DRA-MMCs are well described by equations that involve only the properties of the individual phases. For instance, the density (ρ) of a DRA-MMC can be modeled by a simple rule of mixtures model [2.1]:

\[ \rho_{c} = \rho_{m}v_{m} + \rho_{r}v_{r} \]  

(Eqn. 2.1)

where \( v \) is the volume fraction and the subscripts \( m \) and \( r \) correspond to the matrix and reinforcement, respectively. The density of Al alloys is approximately 2.73 gm/cm\(^2\), while the density of SiC is roughly 3.2 gm/cm\(^2\). Hence, the addition of SiC to Al alloys results in only a modest increase in density.

For continuous fiber Al-MMCs, the modulus of the composite is determined by the orientation of the fibers relative to the stress axis. Figure 2.1 shows the two possible limiting configurations. The composite is assumed to be comprised of alternating layers of \( a \) and \( b \) phases where \( a \) is a ductile matrix and \( b \) is a stiff reinforcement. If a tensile force is applied perpendicular to the layers of fibers, as in Figure 2.1 a, then the stress carried by each phase is the same and is equal to the stress experienced by the composite. This configuration is called the constant stress or isostress model. The composite strain for this case is given by the following equation [2.13]:

\[ \]
\[ \varepsilon_r = V_m \varepsilon_m + V_r \varepsilon_r \]  \hspace{1cm} (Eqn. 2.2)

where \( \varepsilon \) is the strain. \( V \) stands for volume fraction, and the \( m \) and \( r \) subscripts refer to the matrix and reinforcement, respectively. If the deformation in both phases is elastic, the elastic modulus of the composite is given by [2.13]:

\[ E_r = \frac{E_m E_r}{V_m E_r + V_r E_m} \]  \hspace{1cm} (Eqn. 2.3)

where \( E \) is the elastic modulus.

Conversely, if the tensile force is applied parallel to the fiber layers as in Figure 2.1 b, then the strain experienced by both phases is the same and is equal to the composite strain. This arrangement is termed the constant strain or isostrain model. For this model, the composite tensile stress is given by [2.13]:

Figure 2.1: Schematic illustration of (a) isostress and (b) isostrain configurations for a layered composite (reprinted from [2.13]).
\[ \sigma = \sigma_m V_m + \sigma_r V_r \]  
(Eqn. 2.4)

where \( \sigma \) is the stress. The strength of the reinforcement is more efficiently utilized in the isostrain configuration, and this configuration is the most desirable arrangement for a useful composite. If the deformation in both phases is elastic, the elastic modulus of the composite is given by [2.13]:

\[ E_c = V_m E_m + V_r E_r \]  
(Eqn. 2.5)

The elastic modulus of DRA MMCs is bounded by the modulus given by the isostress and isostrain models. Note that both models predict an increase in modulus as the volume fraction of reinforcement is increased. Thus, the addition of a low density reinforcement with a high elastic modulus, such as SiC, to Al alloys provides an appreciable increase in modulus with only a modest increase in weight relative to monolithic Al alloys. One of the utilities of DRA-MMCs lies in this increased stiffness-to-weight ratio (or specific stiffness) with respect to monolithic Al alloys.

Equations for other properties of DRA-MMCs, such as strength, ductility and toughness cannot be accurately described with simple equations involving the properties of the component phases [2.1,2.14]. These properties are also affected by processing and microstructural considerations. Since the reinforcement/matrix interface is typically incoherent, there is no coherency strain field around a particle or whisker to interact directly with dislocations. Strengthening of DRA-MMCs occurs by the increase in forest dislocation density from punching of dislocation loops due to a CTE mismatch between the matrix and reinforcement during heat-treating [2.1]. The amount of strengthening depends on the volume fraction and spacing of the reinforcement. A second advantage of DRA-MMCs accrues from the improved specific strength relative to monolithic alloys.
The increases in specific stiffness and strength of DRA-MMCs are somewhat offset by decreases in ductility and toughness. Ductility and toughness drop as the volume fraction of reinforcement is increased, and both tend to decrease as the average size of the reinforcement increases [2.1]. The toughness and strain to failure are also dependent on the distribution of the reinforcement. Clustering of the reinforcement has been reported to result in a decrease in toughness due to the local development of high levels of constraint relative to other regions [2.1.2.2.14].

2.1.4 Uses of DRA-MMCs

The higher specific strength and specific modulus of DRA-MMCs with respect to monolithic Al alloys make them attractive as structural materials in aerospace and transportation applications, and for use in recreational equipment (bicycle frames, tennis rackets). The structural DRA-MMCs normally contain 10 to 25 volume percent reinforcement. The combination of a lower coefficient of thermal expansion (CTE) and a high specific modulus also provides these materials with excellent dimensional stability which is important in the design of precision equipment, such as optical components.

DRA-MMCs with 40 to 60 volume percent reinforcement have emerged as candidates for microelectronic packaging applications. These composites exhibit the relatively high thermal conductivity typical of aluminum for efficient thermal management and possess a CTE that can be "tuned" by control of the volume fraction of reinforcement to match that of the microelectronic components.
2.1.5 Joining of DRA-MMCs

The effective implementation of DRA-MMCs in engineering structures and devices often mandates that they be welded to both similar and dissimilar materials. As a result, the ultimate utility of these materials hinges on the ability to effectively join them within reasonable economic constraints. To date, relatively little effort has been devoted to evaluating the weldability of DRA-MMCs, and many important issues with respect to the weldability of these materials are not well understood. In fact, the lack of a database on joining of MMCs has been cited as an impediment to their widespread acceptance and use [2.15]. If the advantages of DRA-MMCs are to be exploited on a large scale, then a fuller appreciation of their weldability must be developed. This dissertation will focus on developing joining techniques for SiC-reinforced DRA-MMCs (Al/SiC MMCs) using laser and electron beam welding and on characterizing and understanding the development of microstructure in these welds.

The remainder of the chapter is divided into several major sections. Knowledge of the Al-Si-C ternary system is used in this document as a basis for understanding phase transformations in the welds. Relevant aspects of the Al-Si-C ternary system are discussed in the following section. A review of the Al-Mg-Si system is also given. Some effort has been expended by the casting community in understanding the microstructural development of cast Al/SiC MMCs. The obvious parallels between casting and welding prompt inclusion of a review of this area in a subsequent section. Throughout the review sections, comments of the authors of the papers being reviewed are given in the past tense, while the comments of the student reviewer are typically framed in the present tense.
2.2 The Al-Si-C System

Fusion welding and casting of SiC reinforced Al-MMCs involve melting of the Al alloy matrix with partial or complete dissolution of the SiC reinforcement followed by resolidification and formation or precipitation of a carbide phase(s). This system will be used as a basis for understanding microstructural evolution in the welds made in this study. Hence, a brief review of the phase equilibria and crystallography of the phases of the Al-Si-C system is appropriate at this juncture. Aspects of the three binary systems are discussed first followed by the ternary system. A compilation of the crystallography of the important phases found in the Al-Si-C system is given in Table 2.1 at the end of this section.

2.2.1 The Binary Systems

Information on the Al-Si binary system is well established [2.16]. The Al-Si binary system (Figure 2.2) is a simple eutectic system with two solid solution phases: fcc $\alpha$-aluminum (Fm$\overline{3}m$) and diamond cubic silicon (Fd$\overline{3}m$). Pure Al melts at approximately 660.5° C, while pure Si has a melting point of 1414° C. The eutectic temperature is 577° ± 1° C, and the eutectic composition is 12.2 ± 0.1 at. % Si. The maximum solubility of Si in the $\alpha$ solid solution is 1.5 ± 0.1 at. % at the eutectic temperature, while the solubility of Al in the Si solid solution is less than 0.001 at. %.

Considerably less data are available with regards to the Si-C binary system owing to the difficult experimental requirements. Few comprehensive studies have been undertaken on this system, and contradictory results have been reported. The most widely accepted phase diagram (Figure 2.3) for this system displays a peritectic reaction at 2545° ± 40° C in which SiC forms from solid C and a liquid of 27 at. % C as well as a eutectic reaction at 1404° ± 5° C and 0.75 ± 0.5 at. % C [2.17]. However, another study found the peritectic at 2830° C.
Figure 2.2: The Al-Si binary phase diagram (reprinted from [2.16]).

Figure 2.3: The Si-C binary phase diagram (reprinted from [2.17]).
SiC may exist in several forms. The crystal structure of SiC can be considered as an assembly of corner-sharing tetrahedra. The tetrahedron consists of a carbon atom at the centroid of a tetrahedron with silicon atoms at the corners (or vice-versa). The tetrahedra are connected at their vertices and form double layers of atoms. One layer is made up of carbon atoms and the next layer is comprised of silicon atoms. The crystal structure depends on stacking of the double layers. The cubic form of SiC (β, P43m) is more stable than the hexagonal form (α, P6$_3$mc) at room temperature and atmospheric pressure. The α structure also exhibits several polytypes. Polytypism is a one dimensional disorder that involves structures with different stacking sequences [2.18]. Discussion of polytypes is facilitated by use of the Ramsdell notation which uses a number to describe the number of layers in the repeating cell together with a letter denoting the Bravais lattice. For example, the Ramsdell notation for the β polytype is 3C since the stacking sequence of SiC tetrahedra repeats every three layers along the [111] of a cubic Bravais lattice. The 3C polytype is isostructural with the zinc-blende structure. The 2H structure has a hexagonal lattice that repeats every two layers and is isostructural with the wurtzite structure. By far, the most common hexagonal polytype is the 6H polytype. Schematic depictions of the structures of the 3C and 6H polytypes of SiC are shown in Figure 2.4 [2.19].

Some controversy also exists for the Al-C phase diagram [2.20,2.21]. The phase diagram for this system (Figure 2.5) is characterized by a peritectic reaction between C and a liquid of approximately 27 at. % C to form Al$_4$C$_3$ (R3m). Peritectic temperatures ranging from 1850° C to 2550° C have been reported.

A schematic drawing of the Al$_4$C$_3$ structure is shown in Figure 2.6. The structure of Al$_4$C$_3$ is somewhat more complicated the SiC. The basic unit of structure for Al$_4$C$_3$ is an Al$_2$C double layer of tetrahedra sandwiched between two AlC single layers of tetrahedra.
Figure 2.4: (a) The crystal structure of 3C-SiC; (b) the crystal structure of 6H-SiC. Black atoms indicate C. and gray atoms indicate Si (reprinted from [2.19]).
Figure 2.5: The Al-C binary phase diagram (reprinted from [2.20]).
Figure 2.6: The crystal structure of Al$_2$C$_x$. 
The tetrahedra in the AlC layers point inward toward the Al\textsubscript{2}C double layer. The complete structure is the superposition of three Al\textsubscript{4}C\textsubscript{3} units to give a structure designated 9R using the Ramsdell notation discussed above. The existence of an AlC phase was reported in an early study of this system, but has not been confirmed in subsequent investigations.

2.2.2 The Al-Si-C Ternary System

Several ternary intermediate phases have been reported in the Al\textsubscript{4}C\textsubscript{3}-SiC isopleth (Figure 2.7) [2.21, 2.22]. The existence of an α polymorph and a β polymorph of Al\textsubscript{4}C\textsubscript{3}-SiC (Al\textsubscript{4}SiC\textsubscript{4}) has been reported. Of these two, only the α polymorph (P6\textsubscript{3}mc), which forms via a peritectic reaction at 2080°C [2.21], has been confirmed by later investigators. The diffraction pattern for the β polymorph was subsequently reproduced from a combination of patterns of the α-Al\textsubscript{4}SiC\textsubscript{4} and α'-Al\textsubscript{2}OC suggesting that the β polymorph was incorrectly identified. A second ternary phase Al\textsubscript{4}C\textsubscript{3}·2SiC (Al\textsubscript{4}Si\textsubscript{2}C\textsubscript{5}, R\textsubscript{3}m) was reported to be stable between 1900° and 1970° C. This phase can supposedly be retained to room temperature by rapid quenching, but has not been observed in later studies despite repeated attempts to produce it. Lastly, a 2Al\textsubscript{4}C\textsubscript{3}·SiC (Al\textsubscript{8}SiC\textsubscript{7}, P3) phase has been found to form by a true ternary peritectic reaction at 2085° C [2.21].

Of the ternary compounds reported, only the α polymorph of Al\textsubscript{4}SiC\textsubscript{4} phase and the Al\textsubscript{8}SiC\textsubscript{7} phase are widely accepted to exist. In addition, Al\textsubscript{4}C\textsubscript{3} and SiC are the only confirmed binary compounds. Together with the three pure elements of this system, Al, Si, and C, there are a total of seven elements or compounds found in this system. Accordingly, the base triangle of the Al-Si-C system at temperatures below 577° C where all phases are solid is divided into seven three-phase compatibility triangles as shown in Figure 2.8 [2.21]. The solid phases present at equilibrium below 577° C in each compatibility triangle are given by the apices of the triangles. For example, if a sample of material of the composition
Figure 2.7: The SiC-Al$_4$C$_3$ isopleth (reprinted from [2.22]).

Figure 2.8: Division of the base triangle at low temperatures for the Al-Si-C ternary diagram (reprinted from [2.21]).
indicated by the circle in Figure 2.8 (inside the triangle with apices α-Al, Si and SiC) is equilibrated above the liquidus for this composition and allowed to cool under equilibrium conditions. It will contain α-Al, Si and SiC phases at room temperature.

Information on the phase equilibria during cooling from high temperatures in complicated ternary systems is most easily conveyed with the aid of a liquidus projection. Liquidus projections have been reported for the entire Al-Si-C system (Figure 2.9) [2.22] and for the Al-rich corner (Figure 2.10) [2.23] of the system. The liquidus projection can be used in conjunction with the compatibility triangles to determine the possible solidification paths for cooling of any composition in the ternary system. Completion of the equilibrium solidification path of a sample with a composition inside a given compatibility triangle will take place at the ternary invariant point involving the phases at the apices of the triangle. The seven ternary invariant reactions and the peritectic reaction for the formation of Al₄SiC₄ as well as the invariant reactions for the binary systems are summarized in the reaction diagram of Figure 2.11 [2.23]. The reaction diagram gives all of the possible equilibrium reaction paths for this system.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Bravais Lattice</th>
<th>Space Group</th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>Angle (degrees)</th>
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<td>Fm̅3m</td>
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<td>-</td>
<td>-</td>
<td>90</td>
</tr>
<tr>
<td>Si</td>
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<td>Fd̅3m</td>
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<td>-</td>
<td>90</td>
</tr>
<tr>
<td>Graphite</td>
<td>hex</td>
<td>P63/mmc</td>
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<td>-</td>
<td>6.700</td>
<td>120</td>
</tr>
<tr>
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<td>P63mc</td>
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<td>-</td>
<td>15.117</td>
<td>120</td>
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<tr>
<td>β-SiC</td>
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<td>F4̅3m</td>
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<td>-</td>
<td>-</td>
<td>90</td>
</tr>
<tr>
<td>Al₄C₃*</td>
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<td>R3̅m</td>
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<td>-</td>
<td>24.996</td>
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<td>3.313</td>
<td>-</td>
<td>19.242</td>
<td>120</td>
</tr>
</tbody>
</table>

* using a hexagonal basis

Table 2.1: Crystallographic Information on the Important Phases of the Al-Si-C System
Figure 2.9: Liquidus surface for the Al-Si-C phase diagram (reprinted from [2.22]).

Figure 2.10: Al rich corner of liquidus projection for the stable and metastable Al-Si-C phase diagram (reprinted from [2.23]).
2.3 The Al-Mg-Si System

The matrices of the Al-MMCs studied in this work are casting alloys containing mainly Al, Si and Mg. Alloys based on the Al-Mg-Si system can be strengthened by precipitation-hardening. Consequently, a short review of the physical metallurgy of the Al-Mg-Si system and the crystallography of the phases in this system is useful. A summary of the crystal structures and the lattice parameters of the important phases found in the Al-Mg-Si system is presented in Table 2.2 at the end of this section. As with the review of the Al-Si-C system, the binary systems comprising the ternary system are discussed first followed by the ternary system. Aspects of the Al-Si binary system were considered in the previous section and will not be repeated here. Subsequent discussions will briefly outline important concepts related to precipitation-hardening in the Al-Mg-Si system and precipitation-hardening of Al-MMCs.
2.3.1 The Binary Systems

The binary phase diagram for the Al-Mg system is shown in Figure 2.12 [2.24]. The system displays several eutectic reactions including those at 450° C (~38 at.% Mg) and 437° C (~69 at.% Mg). The maximum solid solubility of Mg in the FCC α-Al phase (Fm3m) is approximately 18.9 at.% at the eutectic temperature of 450° C, while the maximum solid solubility of Al in the HCP Mg phase (P6/mmc) is approximately 11.8 at.% at the eutectic temperature of 450° C. An intermetallic compound (β) with approximate composition Al$_{_{1}}$Mg$_{_{2}}$ (Fd3m) exists below 450° C over the composition range from 38.5 to 40.3 at.% Mg. A second intermetallic compound (γ) with approximate composition Al$_{_{12}}$Mg$_{_{17}}$ (I43m) exists below the congruent transformation at ~455° C over the composition range from 45 to 60.5 at.% Mg. A third intermetallic compound (the R phase, R3) forms from a peritectoid reaction between the β and γ phases at a composition of 42 at.% Mg and a temperature of 370° C.

The binary phase diagram for the Mg-Si system is shown in Figure 2.13 [2.25]. There is very little solubility in the solid solutions at either end of the phase diagram. An intermetallic compound with composition corresponding to Mg$_{_{2}}$Si (Fm3m) exists below 1085° C. There are two eutectics in this system. The first occurs between the HCP Mg phase and Mg$_{_{2}}$Si at 1.16 at. % Si and 637.6 ° C. The second takes place between Mg$_{_{2}}$Si and the Si phase at 53 at. % Si and 945.6 ° C.

2.3.2 The Al-Mg-Si Ternary System

Discussions of precipitation-hardening in the Al-Mg-Si system are facilitated by use of the Al-Mg$_{_{2}}$Si isopleth shown in Figure 2.14 a [2.26]. The Al-rich end of the Al-Mg$_{_{2}}$Si isopleth is presented in Figure 2.14 b [2.27]. Increasing the weight percent of Mg$_{_{2}}$Si allows
Figure 2.12: The Al-Mg binary phase diagram (reprinted from [2.24]).

Figure 2.13: The Mg-Si binary phase diagram (reprinted from [2.25]).
Figure 2.14: (a) The Al-Mg$_2$Si isopleth (reprinted from [2.26]); (b) The Al-rich end of the Al-Mg$_2$Si binary phase diagram (reprinted from [2.27]).
for increased strengths. As shown in Figure 2.14, the maximum solid solubility of Mg\textsubscript{2}Si in the α-Al solution is 1.85%. Most commercial alloys contain between 1 and 1.6 wt. % Mg\textsubscript{2}Si. Al-Mg-Si alloys can be conveniently divided into three categories: 1) stoichiometric alloys: i.e. alloys with an Mg:Si ratio exactly equal to two; 2) alloys with excess Si: i.e. alloys with an Mg:Si less than two; and 3) alloys with excess Mg: i.e. alloys with an Mg:Si greater than two. The volume fraction of Mg\textsubscript{2}Si found in any alloy at equilibrium can be determined by lever-law calculations using the diagram in Figure 2.14. In addition to the Mg\textsubscript{2}Si, the excess Si of type 2 alloys may remain in solution or may form precipitates. Similarly, the excess Mg of type 3 alloys may remain in solution or may form precipitates.

The liquidus projection of the Al-Mg-Si system is given in Figure 2.15 [2.26]. This system is much simpler than the Al-Si-C system discussed earlier, since no ternary compounds exist in the Al-Mg-Si system. In addition to the binary reactions already discussed in sections on binary diagrams, there are seven eutectic reactions and two ternary peritectic reactions. The two eutectic reactions important for the Al-rich alloys of interest here are:

\[ L = \text{Al} + \text{Mg}\textsubscript{2}\text{Si} \]

(Eqn. 2.5)

at the composition corresponding to ~87 at.% Al, ~8 at.% Mg and 593° C as well as

\[ L = \text{Al} + \text{Mg}\textsubscript{2}\text{Si} + \text{Si} \]  

(Eqn. 2.6)

at the composition corresponding to ~81 at.% Al, ~5 at.% Mg and 550° C. The underline refers to a solid solution.
Figure 2.15: The Al-Mg-Si liquidus projection (reprinted from [2.26]).

<table>
<thead>
<tr>
<th>Phase</th>
<th>Bravais Lattice</th>
<th>Space Group</th>
<th>Lattice Parameters</th>
<th>Angle (degrees)</th>
</tr>
</thead>
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<td>Fm̅3m</td>
<td>a (Å)</td>
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<tr>
<td>Mg</td>
<td>hex</td>
<td>P6̅3/mmc</td>
<td>a (Å)</td>
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<tr>
<td>Mg2Si</td>
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<td>Fm̅3m</td>
<td>a (Å)</td>
<td>6.420</td>
</tr>
</tbody>
</table>

Table 2.2: Crystallographic Information on the Important Phases of the Al-Mg-Si System
2.3.3 Precipitation-Hardening in the Al-Mg-Si System

One of the most useful features of Al-Mg-Si alloys is that they can be strengthened by precipitation-hardening as discussed above. For example, the tensile strength of 6061 alloy (1.6% Mg Si) can be increased from approximately 18 ksi to 45 ksi solely by precipitation-hardening heat-treatments. Tensile strengths exceeding 55 ksi are possible with this alloy by cold working prior to heat-treatment.

Precipitation-hardening heat-treatments normally involve a three step process. The first step involves solution heat-treatment at a temperature corresponding to the single phase α-Al region on the phase diagram. This heat-treatment is continued until all of the Mg Si is carried into solution. The samples are subsequently rapidly quenched to retain the Mg Si in solution and finally heat-treated at lower temperatures in the two phase Al-Mg Si region to develop a distribution of fine metastable precipitates based on Mg Si that increase strength by hindering dislocation glide. The time and temperature for the precipitation heat-treatment can be chosen to give maximum strength, maximum corrosion resistance or some combination of the two.

The precipitation heat-treatment results in the formation of a series of intermediate metastable phases culminating eventually in the formation of the equilibrium cubic β-Mg Si phase. The properties depend on the combinations of phases present after heat-treatment. A considerable number of papers have been written on the subject of precipitation-hardening in the Al-Mg-Si system [2.28-2.54]. The discussion presented here derives from a summary of these papers. Unfortunately, there has been considerable controversy and confusion until recently regarding the crystal structure, composition and morphology of the intermediate phase as well as the nomenclature used to describe the intermediate phases. Recent work by Matsuda and coworkers [2.43-2.47] and by Edwards and coworkers [2.48]
has shown that the intermediate phases may possess several different crystal structures and morphologies. The structure and morphology of the intermediate phases appear to depend on several factors including the presence of excess Si [2.39-2.46, 2.52-2.54], the time period between quenching and aging (pre-aging) [2.29, 2.30, 2.34], the temperature of the dwell time before aging [2.29, 2.30, 2.34], the exact aging temperature [2.47], heating rates [2.47], the degree of cold work before aging and the presence of other elements such as Cu.

Throughout the past 35 years, several proposals have been put forth concerning the series of intermediate phases that form during precipitation heat-treatment and their crystal structure. A review of all of the proposed precipitation sequences here is not fruitful and is outside the scope of this work. However, the most recent sequence proposed is as follows: formation of separate clusters of Mg and Si atoms which may also include vacancies (solute/vacancy pairs) → formation of co-clusters containing both Mg and Si (these clusters have also been called GP or GP1 zones in analogy to Al-Cu alloys as well as pre-zones) → formation of small equiaxed precipitates (these precipitates have also been called spherical GP zones) → formation of needle-shaped β'' precipitates (these precipitates have also been called needle-like GP zones) → formation of rod-shaped β' and B' precipitates (β' precipitates form in ternary Al-Mg-Si alloys whereas B' precipitates form in commercial alloys that contain Cu) → formation of the equilibrium β-Mg$_2$Si precipitates [2.48]. Each of these intermediate phases can be viewed as having a metastable solvus line that vary with temperature and composition, and roughly parallel the solvus for the equilibrium β-Mg$_2$Si phase as shown schematically in Figure 2.16. Maximum strength is derived from heat-treatments that favor formation of the needle-shaped β'' precipitates.

As mentioned above, precipitation-hardening in alloys containing excess Si, especially the casting alloys such as those used as the matrix for the Al-MMCs studied in this work, differs somewhat from stoichiometric alloys [2.49-2.51]. The properties of the casting al-
Figure 2.16: A schematic of the metastable solvus lines in the Al-Mg₂Si isopleth.

 alloys are sensitive to quenching conditions. The ultimate tensile strength and the yield strength as well as the ductility have been found to decrease with decreasing quench rates. Lower quench rates result in decreased levels of Si in solution which affect both solution-strengthening and precipitation-hardening processes and in turn lowers the strength [2.51].

2.3.4 Precipitation-Hardening of Al-MMCs

Precipitation-hardening of Al-MMCs has also been found to differ from that of their monolithic counterparts. The overall precipitation sequences of Al-MMCs with Al-Mg-Si alloys matrices has been found to be the same as in monolithic Al-Mg-Si alloys. However, the volume fractions of phases formed for a given temperature as well as the kinetics of formation and dissolution of the intermediate phases were found to differ substantially [2.52-2.54]. Specifically, the kinetics of formation and dissolution of the intermediate phases were found to be accelerated relative to the alloys. The accelerated kinetic rates were
attributed to a decrease in incubation time for nucleation and increase in solute diffusivity (and hence an increase in precipitate growth rates) stemming from an increased dislocation density generated by the large mismatch in CTE between the SiC and Al alloy matrix during quenching.

In addition, the relative amounts of the different intermediate phases were also found to be affected by the presence of the reinforcement. For example, increasing the volume fraction of reinforcement was found to decrease the volume fraction of $\beta$ and $\beta'$ precipitates while increasing the fraction of GP-1 zones (probably Mg/Si/vacancy clusters or $\beta''$ precipitates) [2.54]. The presence of SiC to a 6061 alloy additionally caused the alloy to become quench sensitive. Again, this phenomenon was attributed to the variations in dislocation density due to CTE differences with quenching rates.

2.4 Microstructural Development in Cast Al-MMCs

2.4.1 Reactions Between Al Alloys and SiC

From a thermodynamic standpoint, Al/SiC MMCs are not in chemical equilibrium. Chemical potential gradients exist between the matrix and the reinforcement, and thus, chemical reactions may occur at elevated temperatures. The contact between liquid Al and the SiC reinforcement during joining can lead to the spontaneous formation of an $\text{Al}_4\text{C}_3$ phase according to the following reaction:

$$3 \text{SiC (s)} + 4 \text{Al}_{\text{Al-Si}} = \text{Al}_4\text{C}_3 (s) + 3 \text{Si}_{\text{Al-Si}} \quad (\text{Eqn. 2.7})$$

where the underline indicates solution in the Al-Si alloy matrix. The $\text{Al}_4\text{C}_3$ may form at the Al-SiC interface or may entirely consume the original SiC.
The presence of Al₄C₃ at the Al-SiC interface creates concern regarding diminished material properties for several reasons. The role of the interface region in composites is to allow a uniform transfer of loads between the matrix and reinforcement. The acicular morphology of Al₄C₃ results in stress concentrations which disrupts uniform load transfer and lowers the toughness of the composite. Furthermore, Al₄C₃ readily dissolves in aqueous environments to form CH₄ and porous Al₂O₃ (Al₄C₃ + 6 H₂O = 2 Al₂O₃ + 3 CH₄) that weakens the original interface [2.55]. Considering the necessity for contact between liquid Al and SiC during fusion welding and brazing of Al/SiC MMCs, an understanding of the thermodynamics of Al₄C₃ formation is important. The purpose of this section is to review the thermodynamics of Al₄C₃ formation as reported by several investigators.

Isaikin, Chubarov, Trefilov, Silaev, and Gorelov

The first published thermodynamic analysis of Reaction 2.7 was provided by Isaikin et al. [2.56]. Reaction 2.7 was viewed as the sum of the following reactions where the bracket indicates the liquid Al-Si solution:

\[
\begin{align*}
3 \text{SiC} &= 3 \text{Si}_s + 3 \text{C}_s \quad \text{(Eqn. 2.8)} \\
3 \text{Si}_s &= 3 \text{Si}_l \quad \text{(Eqn. 2.9)} \\
3 \text{Si}_l &= 3 [\text{Si}]_{\text{Al-Si}} \quad \text{(Eqn. 2.10)} \\
4 \text{Al}_l + 3 \text{C}_s &= \text{Al}_4\text{C}_3 \quad \text{s.} \quad \text{(Eqn. 2.11)}
\end{align*}
\]

The ΔGs (in units of calories) for the reactions as reported by Isaikin et al. were:

\[
\Delta G (2.8) = 3 \times (12740 - 0.96 \, \text{T} \ln \text{T} + 0.71 \times 10^{-3} \, \text{T}^2 \\
+ 0.06 \times 10^5 \, \text{T}^{-1} + 4.08 \, \text{T}) \quad \text{(Eqn. 2.12)}
\]
\[ \Delta G (2.9) = 3 \times (11000 - 6.5 T) \]  
\[ \text{(Eqn. 2.13)} \]

\[ \Delta G (2.10) = RT \ln [a]^{3}_{Si} \]  
\[ \text{(Eqn. 2.14)} \]

\[ \Delta G (2.11) = -44000 + 12.4 T. \]  
\[ \text{(Eqn. 2.15)} \]

The \( \Delta G \) for Reaction 2.7 was obtained by summing the \( \Delta G \)s for individual reactions 2.13-2.16.

\[ \Delta G (\text{cal/mole}) = 27220 + 5.14 T - 2.88 T \ln T + 
2.13 \times 10^{-3} T^2 + 1.8 \times 10^4 T^{-1} + RT \ln ([a]^{3}_{Si}) \]  
\[ \text{(Eqn. 2.17)} \]

where \( T \) is the absolute temperature in K, \( R \) is the gas constant, and \( [a]_{Si} \) is the activity of Si in the liquid Al-Si alloy.

At equilibrium, the \( \Delta G(2.17)=0 \), and equation 2.17 was then be solved for \( \ln [a]_{Si} \):

\[ \ln [a]_{Si} = -467 T^{-1} - 0.862 - 0.36 \times 10^{-3} T - 0.30 \times 10^4 T^{-2} + 0.48 \ln T. \]  
\[ \text{(Eqn. 2.18)} \]

Using the relationship between activity of Si and mole fraction Si developed by Mitani and Nagai [2.57], the authors determined the mole fraction of Si in the liquid Al-Si alloy required to prevent the formation of \( \text{Al}_4\text{C}_3 \). They found the mole fraction of Si to preclude \( \text{Al}_4\text{C}_3 \) formation increased from 7.5 % at 1000 K to 17.2 % at 1200 K. They also found that use of an Al alloy already containing amounts of Si near the values for equilibrium prevented formation of any \( \text{Al}_4\text{C}_3 \) on SiC coated filaments, thereby retaining their strength.

Several points concerning the paper by Isaikin et al. should be made. First, the authors appear to be mixing reference states for Si in the calculation. The reference state for Si in Eqn. 2.12 is solid Si, while the reference state used for Si in Eqn. 2.14 is liquid Si. However,
they account for the change in reference state by including the $\Delta G^\circ$ of melting of Si. While this approach is certainly unconventional and adds more steps to the calculation, it is not incorrect.

Next, the expression in given in the paper corresponding to Eqn. 2.18 is incorrect. The coefficient for the $1/T$ term given in the paper is -467. Several possibilities exist for the error: 1) the authors made a math error; 2) there is a misprint in the expression corresponding to Eqn. 2.18; or 3) there is an error or misprint in one or more of the expressions for the $\Delta G$ of the reactions given in Eqns. 2.12 to 2.15. The expressions for the $\Delta G$s for each of the individual reactions 2.12-2.15 were checked, and no mention of thermodynamic data was found in the reference given by the authors (Samsonov) for the $\Delta G$ of formation for $\text{Al}_4\text{C}_3$ (Eqn. 2.11). However, comparison of the equation given for the $\Delta G$ of formation for $\text{Al}_4\text{C}_3$ with equations from other sources suggest that the expression given by the authors is reasonable. Calculations using the data given by the authors give a value of -4566 for the coefficient of the $1/T$ term, and use of this value to determine Si activity gives similar results to those presented by the authors. Hence, the authors used the value (-4566) in their calculations. As this paper was translated from a Soviet journal, the incorrect coefficient in Eqn 2.18 must then stem from a misprint. The correct expression is thus:

$$\ln [a]_{\text{Si}} = -4566 \frac{T}{T} - 0.862 - 0.36 \times 10^{-3} T - 0.30 \times 10^4 T^{-2} + 0.48 \ln T.$$ (Eqn. 2.19)

Even with the correct coefficient for the $1/T$ term, the procedure used by the authors gives only an approximate solution for the problem at hand. Strictly speaking, a fifth reaction should be included to account for the dissolution of pure Al into the Al-Si solution:

$$4 [\text{Al}]_{\text{Al-Si}} = 4 \text{Al}.$$ (Eqn. 2.20)
and the $\Delta G$ for this reaction is:

$$
\Delta G (2.19) = -RT \ln [a]^4_{Al} . \tag{Eqn. 2.21}
$$

Summation of the $\Delta G$s for all of the reactions including the $\Delta G$ for Eqn. 2.19 yields:

$$
\Delta G (\text{cal/mole}) = 27220 + 5.14 T - 2.88 T \ln T + 2.13 \times 10^{-3} T^2
+ 1.8 \times 10^4 T^{-1} + RT \ln ([a]^3_{Si}) - RT (\ln [a]^4_{Al}) . \tag{Eqn. 2.22}
$$

Again, when reaction 2.7 is in equilibrium, the total $\Delta G$=0. The resulting equation cannot be solved directly for the activity of Si since it contains an expression relating the natural logarithm of the ratio $[a]^3_{Si} / [a]^4_{Al}$ to temperature:

$$
\ln \left( \frac{[a]^4_{Al}}{[a]^3_{Si}} \right) = 1.370 \times 10^4 - 1.449 \ln T + 1.072 \times 10^{-3} T + 9.059 \times 10^3 T^{-2} + 2.587 
$$

**(Eqn. 2.23)**

To simplify the calculation of the activity of Si, the authors assumed that the volume fraction of Al in the alloys of interest is often greater than 0.9, and that large errors do not occur if the activity of Al is close to one. If the Al activity in Eqn 2.23 is set to one, then Eqn. 2.19 will result. However, note that the activity term for Al is raised to the fourth power, and that the results of the authors calculation show that the volume fraction of Al at equilibrium decreases with increasing temperature to a value less than 0.83. This assumption is expected to introduce some error to the result.

Two final points should be made concerning the paper by Isaikin et al. The activity for Al that they calculate is based on liquid Si as the reference state. However, the data for the variation of Si activity with mole fraction Si reported by Mitani and Nagai [2.57] and used to find the mole fraction of Si at equilibrium appears to be based on solid Si as the reference state.
state. The data of Mitani and Nagai can be modified to account for the difference in reference state, but the authors do not make this modification. Finally, the calculation assumes that the liquid solution contains only Al and Si, and the activity of C in the liquid solution is ignored. Both of these oversights likely introduce some error to the result.

Iseki, Kameda, and Maruyama

A study of the thermodynamics and kinetics of Reaction 2.7 in the temperature range from 700° C to 1200° C has been carried out by Iseki et al. [2.58]. They reacted Al powders of 99.5 wt. % purity with SiC powders of three different grain sizes in a molar ratio of 3:4. The powders were pressed into a green body and heat-treated at temperatures ranging from 700° C to 1200° C at a pressure of 10^-2 Pa for 15 to 180 minutes. In order to study the effects of free silicon on Reaction 2.7, similar experiments were conducted on samples produced by adding 1 wt. % or 10 wt. % Si powder to the Al powder. After heat-treatment, the samples were ground into a fine powder, and the amount of Al$_4$C$_3$ formed was determined by quantitative analysis using X-ray diffraction (XRD).

The extent of Al$_4$C$_3$ formation, $\alpha$, was determined from the stoichiometry of Reaction 2.7. Specifically, the components were present in the heat-treated samples in the following ratio SiC: Al: Al$_4$C$_3$: Si = 3(1-\alpha): 4(1-\alpha): 3\alpha: 3\alpha. The term $\alpha$ may be viewed as the progress variable for Reaction 2.7. For example, for each mole of Al$_4$C$_3$ formed, three moles of Si were produced, while three moles of SiC and four moles of Al were consumed. Values of $\alpha$ were determined from changes in the X-ray intensity for the respective diffraction peaks. Note that this approach assumes that the differences in X-ray absorption coefficients for the four components do not greatly affect the intensities of the different peaks when the ratio of the components was changed. No mention was made of a correction procedure to account for the possible errors associated with this assumption. This oversight likely introduced some error in the results.
The experimental results of this work demonstrated that the rate of Al₄C₃ formation (α) dropped dramatically after a period of 30 minutes to an hour depending on the original SiC grain size, as shown in Figure 2.17. Note that the amount of Si produced (3α) also decreased at the same time. The results also illustrated that the saturated value of α was greater at higher temperatures, and that α decreased as a function of the amount of free Si powder added to the Al powder.

Iseki [2.58] transformed the data for free energy provided by Isaikin et al. [2.56] from units of calories to Joules and used a procedure identical to that of Isaikin et al. to derive the following expression for the Gibbs free energy (ΔG) of equation 2.7:

\[
\Delta G (J/mol) = 113900 - 12.06T \ln T + 8.92 \times 10^{-3} T^2 \\
+ 7.53 \times 10^{-4} T^{-1} + 21.5 T + 3RT \ln a_{Si} \tag{Eqn. 2.24}
\]

where T is the absolute temperature in K, R is the gas constant, and \( a_{Si} \) is the activity of Si in the liquid Al-Si alloy. This expression indicated that the free energy of formation of Al₄C₃ increased (i.e. a smaller negative value) for increases in the activity of Si.

Note that the equation reported in the original paper by Iseki and coworkers contains a misprint. Calculations using the data from Isaikin et al. give a value of 7.53 \times 10^{-4} for coefficient of the T⁻¹ term. This oversight is corrected in a second paper by the same workers [2.59]. Thus, the correct expression is:

\[
\Delta G (J/mol) = 113900 - 12.06T \ln T + 8.92 \times 10^{-3} T^2 \\
+ 7.53 \times 10^{-4} T^{-1} + 21.5 T + 3RT \ln a_{Si} \tag{Eqn. 2.25}
\]

Despite the misprint, calculations were performed correctly.
As Reaction 2.7 proceeded at a given temperature, the increase in the amount of Si in the melt resulted in an increase in the activity of Si which caused the Gibbs free energy for the reaction to become less negative. The Si activity eventually increased to the point where the free energy equaled zero ($\Delta G = 0$), and the reaction attained equilibrium. Equation 2.25 was used to determine the activity of Si at equilibrium (i.e. when $\Delta G = 0$).

Utilizing data for the Si activity as a function of mole fraction Si ($= 1 -$ mole fraction Al) determined by Mitani and Nagai [2.57], Iseki et al. then determined a relationship between temperature and the saturated value for $\alpha$ at equilibrium. The plot of $\alpha$ at equilibrium vs. temperature is given in Figure 2.18. The value $\alpha$ corresponding to the saturation of Reaction 2.7 at 1273 K ($1000^\circ$ C) was determined to be $= 0.22$. This value of $\alpha$ corresponds to a molar fraction of Si of $= 17.5\%$. Not surprisingly, this value correspond closely with the value found by Isaikin et al. at 900$^\circ$ C. When 10 wt. % Si was added to the Al powder, the calculated value for $\alpha = 0.095$. The lower value of $\alpha$ indicated that the reaction did not proceed as far when free Si was added.

Note the method used by Iseki et al. was identical to that employed by Isaikin et al and that all of the points made earlier concerning this method are applicable to the discussion above.

Handwerker, Vaudin, Kettner, and Lee

Handwerker et al. produced SiC/Al composites by mixing Al powders of 99% purity with SiC powder in a 1:1 molar ratio [2.60]. The SiC powder was a combination of $\alpha$ (hexagonal) and $\beta$ (fcc) crystal structures. The mixed powder composites were sealed in a crucible, heat-treated for nine days at 920$^\circ$ C, and air-quenched to room temperature. The resultant microstructures were examined using optical microscopy, electron probe microanalysis (EPMA), and transmission electron microscopy (TEM).
Figure 2.17: Extent of Al4C3 reaction (\( \alpha \)) as a function of time at 1273 K (1000° C) (reprinted from [2.58]).

Figure 2.18: Relation between saturated value of extent of reaction (\( \alpha \)) as a function of temperature (reprinted from [2.58]).
Reaction 2.7 was identified as the principal reaction that occurred during heat-treatment of these composite samples. Although no kinetic measurements were discussed in this paper, the formation of $\text{Al}_4\text{C}_3$ was reported to occur via two mechanisms. The operative mechanism appeared to depend on the crystallographic orientation of the SiC relative to the Al alloy melt. For SiC basal planes (i.e. (0001)) in contact with the melt, the reaction involved the dissolution of SiC and diffusion of C (and Si) through the liquid alloy to form $\text{Al}_4\text{C}_3$ at the liquid/$\text{Al}_4\text{C}_3$ interface. Precipitates of $\text{Al}_4\text{C}_3$ reportedly formed at preferred sites on the (0001) surfaces (possibly screw dislocations or other defect sites), while nearby areas on the exposed (0001) plane initially resisted attack before dissolving. The presence of dislocations and twins in the SiC and $\text{Al}_4\text{C}_3$ determined from TEM analysis were also reported by the authors of this study. A similar dissolution/precipitation reaction has also been reported by Viala et. al [2.23]. Attack of the SiC by Al resulted in the development of facets and ledges on the SiC.

For planes of non-basal orientation, the reaction proceeded by counterdiffusion of Si, C and/or Al through the $\text{Al}_4\text{C}_3$ to produce $\text{Al}_4\text{C}_3$ at the liquid/$\text{Al}_4\text{C}_3$ interface. The second reaction mechanism was substantiated by the identification of isolated SiC platelets embedded in the $\text{Al}_4\text{C}_3$ layer. The orientation of the SiC platelets relative to the $\text{Al}_4\text{C}_3$ was identical to that of the bulk SiC suggesting that the platelets were once continuous with the underlying SiC and that partial transformation of the SiC to $\text{Al}_4\text{C}_3$ had occurred by diffusion through the $\text{Al}_4\text{C}_3$.

Isothermal sections of the Al-Si-C ternary phase diagram from 500°C to 1500°C were also calculated by Handwerker using the available thermodynamic data. The isothermal section at 900°C was used to interpret the microstructures of the SiC/Al composites used in their study. The starting composition fell within the compatibility triangle defined by $\text{Al}_4\text{C}_3$. [39]
SiC, and an Al-18 at.% Si alloy. This suggests that at equilibrium the mole fraction of Si in the liquid Al-Si alloy is \( \sim 18\% \). Note that this value agrees very well with the equilibrium values found by Isaikin et al. [2.56] and Iseki et al. [2.58,2.59]. However, the EPMA of the Al-Si alloy showed that a final Si content of only 13 at. %. The authors concluded that equilibrium had not been attained and that the microstructure and phase distribution for this heat-treatment were controlled by kinetic considerations rather than by thermodynamic considerations.

**Romero and Arsenault**

Romero and Arsenault produced Al/SiC/20p composites by blending Al 1100 powders with \( \alpha \) (hexagonal) SiC powder [2.61]. Different sets of samples were heat-treated at 590° C and 680° C for various times to compare the effects of the contact of SiC with solid and liquid Al. Samples were also apparently heated to 700° C and above for different times. After heat-treating, the samples were examined using TEM and high resolution electron microscopy (HREM). No change was observed in the interface of the samples heated to 590° C. This result suggests that Reaction 2.7 proceeds very slowly in the solid-state. Substantial dissolution of SiC along the basal planes was noted for the samples heated to 680° C. The surface of the SiC developed serrations and cracks during reaction with the liquid Al leading to penetration of Al along channels formed by the serrations. No Al₄C₃ was observed to have formed at temperatures below 700° C. Below 700° C, SiC was thought to dissolve from high energy sites along the interface and reprecipitate at low energy sites without the formation of Al₄C₃. Above 700° C, a continuous layer of Al₄C₃ was found covering the surface of the SiC.

**Oden and McCune**

The preceding papers involved studies of phase stabilities at relatively low temperatures. Fusion welding can often involve peak temperatures above 1500° C, and some dis-
cussion about higher temperature is appropriate for comparison to welding situations. Oden and McCune investigated phase equilibria in the Al-Si-C system at 2000° C and 2150° C [2.21]. They heated sealed crucibles containing samples of various compositions for 60 minutes at 2000° C and 15 minutes at 2150° C. Other samples were heated to 2000° C for various times to determine the time for saturation of an Al-50% Si with C. Followingheat-treatment, the samples were examined using optical microscopy (OM), XRD and EPMA.

In addition to Al₄C₃, ternary carbide phases stable to their peritectic decomposition temperatures at 2085° C and 2080° C were observed with compositions 2Al₄C₃·SiC (Al₈SiC₇) and Al₄C₃·SiC (Al₄SiC₄) respectively. The Al₄C₃ and Al₄SiC₄ exhibited a needle-like morphology. Isothermal sections for the Al-Si-C diagram were constructed at 2000° C and 2150° C, see Figures 2.19 a and b. No evidence of Al₄C₃·2SiC (Al₄Si₂C₅) was found in this work despite repeated attempts to produce it. The authors developed a partial liquidus projection that was consistent with their results and formulated the reaction diagram of Figure 2.11 to describe the possible binary and ternary reaction paths.

Viala, Fortier and Bouix

Viala et al. studied stable and metastable phase equilibria between Al-Si alloys and SiC at temperatures up to 1900 K (1627° C) [2.23]. They constructed isothermal sections with tie lines to describe phase equilibria of the system at temperatures of 840 K (567° C), 1270 K (997° C) and 1770 K (1497° C) as well as a liquidus projection shown earlier in Figure 2.10. The isothermal projections at 1270 K and 1770 K are shown in Figures 2.20 a and b. Samples of pure Al and SiC heated to 840 K for long periods of time showed no evidence of reaction. The Al alloy matrix/SiC interface of these samples remained smooth and distinct. Similar samples heated to 1150 K and 1270 K rapidly acquired a jagged reaction layer of Al₄C₃ along the interface. However, samples containing a mixture of an Al-20 at. % Si alloy and SiC heated to 1270 K were free from attack. Finally, samples of pure Al and SiC heated to 1770 K were observed to contain needles of Al₄SiC₄.
Figure 2.19: (a) Al-Si-C isothermal section at 2000° C; (b) Al-Si-C isothermal section at 2150° C (reprinted from [2.21]).
Figure 2.20: (a) Al-Si-C isothermal section at 1270 K (997° C);
(b) Al-Si-C isothermal section at 1770 K (1497° C) (reprinted from [2.23]).
The authors also developed a model to describe the series of chemical interactions between Al and SiC from room temperature to 1900 K (1627° C). These models and diagrams and those of Oden and McCune (Figures 2.14 a and b) provide a starting point for understanding microstructural development in welds of Al/SiC MMCs.

2.4.2 Reinforcement Redistribution During Solidification

While the mechanisms of solute redistribution at an advancing S/L interface are well established, little is known about the effects of insoluble particles on the interface. When a moving S/L interface intercepts an insoluble particle, it can either push or engulf the particle. Engulfment occurs when the solid grows around the particle. If the particle is not engulfed, it can be pushed and subsequently entrapped along a cell boundary when two or more S/L interfaces converge. Entrapment of particles along cell boundaries can lead to particle clustering and a decrease in toughness as discussed earlier [2.1,2.2,2.14].

A number of theoretical models have been proposed to describe redistribution of reinforcement during solidification. They can be broadly classified in to three general categories: 1) thermodynamic models, 2) kinetic models, and 3) models involving thermal properties. The thermodynamic models invoke a balance of surface free energies, while the kinetic models involve surface energy driven diffusion and fluid flow. Models in the third category consider the effects of the particle on the thermal fields ahead of the S/L interface. Several of the models are reviewed in this section. Many details of the derivations are omitted for the sake of brevity.

Omenyi and Neumann (O&N)

Omenyi and Neumann reported that thermodynamic effects will dominate if the solidification rate is slow and the S/L interface is planar [2.62]. They assumed that the criterion
for engulfment of a spherical particle involved a net decrease of Helmholtz free energy. If $\Delta F_{\text{net}} < 0$, then the particle will be engulfed, and if $\Delta F_{\text{net}} > 0$, the particle will be rejected and pushed. $\Delta F_{\text{net}}$ for engulfment of a spherical particle of unit surface area is given by:

$$\Delta F_{\text{net}} = \gamma_{\text{ps}} - \gamma_{\text{pl}}$$

(Eqn. 2.26)

which is simply the free energy of the final position minus that of the initial position.

**Uhlmann, Chalmers, and Jackson (UC&J)**

The model suggested by Uhlmann et al. assumed that the pushing of a single particle by an advancing interface requires both a force preventing engulfment of the particle and feeding of liquid to the region between the particle and interface [2.63]. They proposed that surface energy effects provide the repulsive interaction. Furthermore, they assumed that the surface free energy varies as a function of distance between the particle and the interface according to the following relation:

$$\Delta \sigma = \Delta \sigma_0 \left(\frac{d_0}{d}\right)^n$$

(Eqn. 2.27)

where $d$ is the distance between the particle and the interface, $d_0$ is the minimum possible separation between the particle and the interface, and $n$ is a constant on the order of 4 or 5. Note that $\Delta \sigma_0$ decreases rapidly for large $d$ and increases rapidly for small $d$. $\Delta \sigma_0$ is the surface free energy at $d_0$ and is given by:

$$\Delta \sigma_0 = \sigma_{\text{sp}} - (\sigma_{\text{sl}} + \sigma_{\text{lp}}).$$

(Eqn. 2.28)

The UC&J model introduced the concept of a critical velocity for engulfment. The relation for the critical velocity was derived from an equation of conservation of mass:

$$v_c = \frac{1}{2(n+1)} \left(\frac{L_0 V_0 D}{KTR^2}\right)$$

(Eqn. 2.29)
where: $L$ is the volume latent heat of fusion, $a_0$ is a constant from the Gibbs-Thomson relation and is of the order of an atomic diameter, $V_0$ is the atomic volume. $K$ is the Boltzmann constant. $T$ is absolute temperature, and $1/R$ is the equilibrium curvature of the interface.

**Bolling and Cisse (B&C)**

Bolling and Cisse developed a model that computes the interface shape by considering kinetic undercooling and shifts in the melting point due to interface curvature [2.64]. The particle is initially assumed to be separated from the interface. Subsequently, a small force (gravity or viscous drag) acts on the particle and promotes contact. The particle must then be accelerated in the direction of interface motion until a steady state configuration is reached between the particle and the S/L interface. The critical velocity for engulfment based on the B&C model is given by the following expression:

$$v_c = \left( \frac{4\psi(\alpha, \beta) KT \gamma_{sl} a_0}{9\eta r^2} \right)^{1/2}$$

(Eqn. 2.30)

where: $\psi(\alpha, \beta)$ is a function of the interface shape and characteristics of the melt and properties. $K$ is the Boltzmann constant. $T$ is absolute temperature. $\eta$ is the viscosity of the liquid. $\gamma_{sl}$ is the volume S/L surface free energy. $r$ is the particle radius. and $a_0$ is the atomic spacing. Note that the symbol $R$ is used in this model to denote particle radius, while in the previous model $R$ is used to describe interface curvature.

**Zubkov, Lobanov, and Nikonova (ZL&N)**

Zubkov et al. proposed an empirical relation for prediction of engulfment based on the ratio of the thermal conductivity of the particle to that of the liquid [2.65]. According to this model, if $\lambda_p/\lambda_l < 1$ (Eqn. 2.31), the interface growth rate increases and a protuberance appears on the interface that pushes the particle (where $\lambda$ is the thermal conductivity and $p$
and 1 refer to the particle and liquid, respectively). Conversely, if $\lambda_p/\lambda_l > 1$ (Eqn. 2.32), the interface growth rate decreases, and a depression develops on the interface that envelops the particle.

**Rohatgi and Surappa (R&S)**

Rohatgi and Surappa proposed an empirical relation similar to that of the ZL&N model based on the ratio of heat diffusivities of the particle and the liquid \[2.66\]. This model predicts engulfment if the following criterion is met:

$$\frac{\lambda_p}{\lambda_l} \left( \frac{C_p}{C_l} \frac{\rho_p}{\rho_l} \right)^{1/2} > 1$$  \hspace{1cm} (Eqn. 2.33)

where $\lambda$ is the thermal conductivity, $C$ is the heat capacity, $\rho$ is the density, and $p$ and $l$ refer to the particle and liquid, respectively. When the heat diffusivity of the particle is greater than that of the liquid, a depression forms on the interface that engulfs the particle. When the heat diffusivity of the particle is less than that of the liquid, a mound is formed on the interface which pushes or rejects the particle.

**Stefanescu, Dhindaw, Kacar and Moitra (SDK&M)**

More recent attempts have been made to develop models that incorporate both kinetic and thermal effects. One such model has been reported by Stefanescu et al \[2.67, 2.68\]. The expression for the critical velocity is:

$$v_c = \frac{\Delta \sigma \rho d_0}{6(\eta - 1) \eta_f (2-k_p/k_l)}$$  \hspace{1cm} (Eqn. 2.34)

where $k$ refers to thermal conductivity. The effect of many particles can be included in this model using Einstein’s equation for relative viscosity \[2.69\]:

$$\eta^* = \eta (1 + 5v_f/2)$$  \hspace{1cm} (Eqn. 2.35)
where $\eta^*$ is the effective viscosity and $\nu_f$ is the volume fraction of particles. This expression may be used in Equation 2.33 to determine the critical velocity for a composite with a given volume fraction of reinforcement.

Rohatgi, Asthana and Yarandi

A comparison of calculated values for $v_C$ for an A357/SiC/15p (20 µm SiC particles) has been reported by Rohatgi et al. [2.70]. The O&N model (Eqn. 2.26) predicts engulfment for SiC particles of all sizes. A critical velocity cannot be determined using this model. The theoretical critical velocity for a 20 µm SiC particle calculated using the UC&J model (Eqn. 2.29) is 0.187 µm/sec. For the B&C model (Eqn. 30), the theoretical critical velocity calculated for a 20 µm SiC particle is 4.24 µm/sec. All three of the thermal property criterion models discussed predict engulfment for 20 µm SiC particles. Again, a critical velocity cannot be determined using these models. Finally, the combined kinetic and thermal model of SDK&M (Equation 2.34) predicts a critical velocity of approximately 2500 µm/sec for 20 µm particles of SiC.

Rohatgi et al. have also determined the critical velocity for an A357-SiC composite with 15 volume percent of 20 µm SiC particles from a unidirectional solidification experiment using a Bridgeman furnace [2.70]. They found a critical velocity of 0.377 µm/sec for these conditions. Lloyd and Chamberlain studied dendrite arm spacing (DAS) and particle distribution as a function of cooling rate in castings of an A356-SiC composite with 15 volume percent of 15 µm SiC particles [2.8] (note that cooling rate is not the same as interface growth rate, though the two are related). They found that particles were pushed to interdendritic regions and trapped over a wide range of cooling rates. While they did not actually measure interface growth rates, it is likely that the growth rates exceeded the critical velocity of 0.377 µm/sec found by Rohatgi et al.
Presently, no one model appears capable of fully describing a criterion for engulfment for all systems. The predictions based on different models vary and are often contradictory. The UC&J model predicts a critical velocity on the order of that found in the unidirectional solidification experiments of Rohatgi et al. [2.70], but underestimates the critical velocity for the castings. The discrepancies likely arise from the fact that the UC&J model assumes a planar interface, neglects thermal effects, and was derived for a single particle. Note that the prediction of the B&C model is not consistent with the experimental results for the unidirectional solidification experiments. The difference between the measured and calculated values for this model likely stem from the inability to accurately define \( d_0 \) and \( n \). In addition, the B&C model was derived for a single particle, and the effects of multiple particles were ignored. The SDK&W model is likely more applicable to the welds and castings.

The accuracy of models which include the effects of surface energies suffer from the inability to obtain accurate and meaningful values for the surface energies at the appropriate temperatures. The surface energy values found using sessile drop experiments are equilibrium values and are not necessarily applicable to moving interfaces. The equilibrium values also do not apply when chemical reactions occur at the reinforcement/liquid interface during melting. For example, the contact between liquid Al and the SiC reinforcement during melting can lead to the spontaneous formation of \( \text{Al}_4\text{C}_3 \) as discussed earlier. Formation of \( \text{Al}_4\text{C}_3 \) at the reinforcement/liquid interface will likely result in a surface energy that changes with time as the reaction proceeds. Models which include the surface energy effects are likely more accurate when the particle is close to the S/L interface and do provide a method for determining a critical velocity. Models that use thermal property criterion for engulfment do not provide a means of calculating a critical velocity, but use physical properties that are more easily obtained. These models appear to better describe interactions when the
particle is far from the S/L interface. A general functional relationship to fully describe a critical velocity should consider thermodynamic and kinetic effects, as well as thermal properties.

2.4.3 Grain Size vs. Cooling Rate

Grain size, or cell size in the case of cast materials, is an important microstructural feature. The effect of grain size on the yield strength of the material can be given by the well known Hall-Petch relation [2.71]:

$$\tau = \tau_0 + k d^{1/2}.$$  \hspace{1cm} (Eqn. 2.36)

where $\tau$ is the applied shear stress at which dislocation activation occurs, $\tau_0$ is the intrinsic shear stress of the grain and $d$ is the grain diameter. Hence, the yield strength of a material increases as the grain size decreases.

Lloyd investigated the variation in solidification cell size with cooling rate for Al/SiC MMCs and unreinforced alloys [2.72]. Composite and alloy melts were poured at 675°C into a one inch diameter mold with a chill at the bottom end. Thermocouples were placed at several positions in the mold to measure cooling rates as a function of position. This arrangement produced cooling rates ranging from roughly 1 to 50 °C/s. The cell size $d$ depends on the cooling rate $R$ according to the following relation [2.73]:

$$d R^a = \text{constant.}$$  \hspace{1cm} (Eqn. 2.37)

The value of $a$ for Al alloys was found to range from 0.34 to 0.39 which was consistent with previous results. With the exception of the MMCs with a 6061 matrix, the presence of 10 to 20 vol. % SiC roughly 10 μm in size did not significantly alter the value for ‘$a$’ over the
cooling range used in this work. The 6061/SiC MMCs exhibited a larger value of \( a \) relative to the monolithic 6061 alloys (0.38 vs. 0.32) indicating a slight refinement in cell size.

The results of the work on cell size vs. cooling rate and reinforcement redistribution indicate that faster cooling rates promote finer grain sizes and more uniform reinforcement distributions as seen in Figure 2.21. Thus, manipulation of cooling rate offers a method for control of microstructure and properties.

2.5 Summary

Several issues pertinent to welding of aluminum metal-matrix composites (Al-MMCs) have been reviewed. The first section of the review provided an introduction to the production, properties and uses of Al-MMCs. In the second and third sections, phase equilibria in the Al-Si-C and Al-Mg-Si systems were reviewed along with the crystallography of the phases found in these systems. In the fourth section, issues of microstructural development in cast Al-MMCs were discussed including: (1) the thermodynamics of reactions between liquid Al alloys and SiC; (2) reinforcement redistribution during solidification of Al-MMCs; and (3) grain size vs. cooling rates for Al-MMCs. The effects of the topics discussed in this chapter and a review of papers concerned directly with joining of Al-MMCs is provided in the next chapter.
Figure 2.21: Cooling rate dependence of cell size in A356 materials (reprinted from [2.72]).
In the previous chapter, process/structure/property relations for Al-MMCs were discussed. In the first part of this chapter, the information developed in the last chapter is applied to the case of welding of Al-MMCs. Subsequently, a review of papers concerning welding of Al-MMCs is presented.

3.1 Welding Concerns

Several factors govern the weldability of Al/SiC MMCs. As with all aluminum alloys, appropriate cleaning of the parts before joining and proper shielding during joining are necessary to avoid hydrogen contamination and the subsequent formation of porosity. However, the weldability of Al/SiC MMCs differs markedly in several respects from that of monolithic Al alloys due to the presence of the reinforcement. The addition of SiC to the Al matrix acts to modify several physical and chemical properties, which in turn greatly affect the weldability. Thus, an appreciation of the weldability of Al/SiC MMCs is based upon a knowledge of the effects of the various matrix/reinforcement interactions. Some of these issues are reviewed below.

As discussed in the previous chapter, Al/SiC MMCs are not in thermodynamic equilibrium. The contact between liquid Al and the SiC reinforcement during joining may lead to
the spontaneous formation of $\text{Al}_4\text{C}_3$ and other carbide phases as discussed earlier. The attack of SiC by Al has been shown to result in roughened and serrated interfaces as well as the formation of acicular carbide phases. The role of the interface region is to facilitate smooth load transfer between the matrix and the SiC. The sharp angles of the jagged and serrated interfaces and the needle-like morphology of the reaction products create concerns regarding stress concentrations and possible reduced toughness of the MMC.

The weld pool of the Al-MMC will generally contain a solid constituent, since the reinforcement does not usually melt or completely dissolve during welding. Thus, the weld pool of an Al/SiC MMC can be considered as a two-phase liquid/solid mixture in which the liquid consists of a continuous network. The most obvious effect of the presence of the solid reinforcement is the increased viscosity of the weld pool. Suspension of spherical solid particles in liquids is known to increase viscosity of the suspension [2.69, 3.1-3.6]. Increased viscosity of weld pools in Al-MMCs has been cited as a problem by several welding researchers.

The presence of the solid SiC also affects heat transfer in the weld area. The presence of SiC with its lower thermal diffusivity relative to Al decreases the composite thermal diffusivity. Additionally, the SiC influences convective heat transfer. In the weld pool of a monolithic alloy, convective heat transfer dominates over conductive heat flow, and the pool temperature is relatively uniform. Since mass transfer is limited by the viscous nature of the MMC weld pool, convective heat flow in the MMC weld pool is probably less effective than in Al alloys. As a result, conductive heat flow through the Al alloy matrix is thought to determine the temperature distributions and cooling rates in and around the weld pool of Al-MMCs. The differences in heat flow in the MMC relative to monolithic Al alloys can result in different microstructures and the stress distributions in the MMC weld.
Another type of matrix/reinforcement interaction occurs during solidification of DRA MMCs. Under certain conditions, the SiC reinforcement may be pushed ahead of the advancing solidification front, as discussed in Chapter 2 [2.62-2.70]. Since the reinforcement is pushed to the last areas to solidify, its distribution is governed by solidification substructure (cell and dendrite) size. As mentioned earlier, clustering of the SiC along cell boundaries, and especially at triple points, can result in a decrease in toughness due to the local development of high levels of constraint relative to other regions [2.1, 2.2, 2.14].

The collection of reinforcement in front of the advancing solid/liquid interface may also locally hinder heat transfer and solute diffusion ahead of the interface. The modification of thermal and solutal fields ahead of the solid/liquid interface likely affects solidification conditions. Both the pushing of reinforcement and modification in solute segregation will affect the final weld microstructure.

3.2 Joining of Al/SiC MMCs

3.2.1 Process/Microstructure Relations for Joining of Al/SiC MMCs

The materials scientist is interested in the effects of processing on the resultant microstructure and properties of a material. The present application is concerned with the effects of joining processes on the microstructures and properties of Al/SiC MMCs. Ideally, the joining process would have no effect on the properties, however this rarely occurs. In practice, joining techniques should be chosen to limit undesirable microstructures and to promote desirable microstructural features.

The effects of several microstructural features were introduced and discussed in the last chapter. These effects are likely also be important with respect to joining of Al/SiC MMCs.
The attack of the SiC reinforcement by liquid Al has been shown to result in roughened and serrated interfaces as well as faulting of the SiC and the formation of acicular carbide phases [2.21, 2.23, 2.56, 2.58-2.61]. To minimize losses in toughness, joining methods should be tailored to limit the attack of the SiC. In addition, joining techniques should be engineered to limit decreases in toughness due clustering of the SiC reinforcement during solidification [2.1, 2.2, 2.14]. Joining processes that promote fine grain size and thus increased yield strength are also desirable. The presence of hydrogen porosity in Al products tends to lower the weld joint mechanical properties and should be avoided through the use of proper cleaning and shielding. Finally, the use of monolithic filler metals during welding may result in a decrease in the volume fraction of reinforcement in the weld joint and a commensurate reduction in the joint stiffness. Truly composite joints await the development of composite filler materials.

3.2.2 Methods for Joining of Al/SiC MMCs

Several types of processes have been used to join Al/SiC MMCs. Unfortunately, there is no single ideal or perfect joining process for Al/SiC MMCs. Each type of process has inherent advantages and shortcomings as discussed below. A brief review of the welding processes referred to in this section is provided in Appendix A.

Like the methods of manufacture, the methods for joining of Al/SiC MMCs can be broadly divided into three categories according to the physical state of the matrix during joining: solid-state, liquid-state and two phase. Solid state processes such as friction welding (FRW) do not entail melting of the matrix alloy and consequently do not suffer from the problems associated with reinforcement redistribution during solidification or chemical reactions between the matrix and SiC [3.7-3.12]. FRW is capable of producing quality welds in Al-MMCs with high joint efficiencies [3.13-3.14]. Joint efficiency is defined as the ratio of
the tensile strength of a weld sample to that of the parent material.) However, the high stresses associated with friction welding can result in crushing or cracking of the SiC reinforcement, and the process is presently limited to cylindrical geometries.

Diffusion bonding is another solid state joining process that has been used with Al-MMCs [3.15-3.18]. The stresses associated with diffusion bonding are lower that those found in FRW, but the thermal cycles are normally much longer. Diffusion bonding involves heating of the whole assembly to be joined to a high homologous temperature under high pressure. This can result in a loss of strength in strain-hardened alloys (due to recovery and recrystallization) as well as precipitation-strengthened alloys (due to dissolution or overaging of precipitates). Problems with reinforcement clustering or loading near the joint interface have also been reported [3.15-3.18].

Two phase (solid/liquid) processes like brazing and transient liquid phase (TLP) bonding involve melting and spreading of a filler metal with a liquidus temperature below that of the matrix alloy between the parts to be joined [3.19, 3.20]. As a result of interdiffusion with the filler metal, small amounts of the matrix alloy dissolve and mix with the filler metal. Unfortunately, brazing and TLP bonding also typically involve heating of the whole assembly to be joined to just below the solidus temperature of the matrix alloy. As with diffusion bonding, heating to these temperatures can result in a loss of strength in strain hardened and precipitation strengthened alloys. The presence of Kirkendall porosity [3.20] and increased volume fraction of reinforcement near the joint interface have also been observed during TLP bonding.

Fusion welding involves melting of the matrix with a heat source such as an arc or a high energy beam. A weld pool is produced by the heat source and is carried along the joint
interface between the pieces to be joined to effect the weld. Melting occurs along the front surface of the moving weld pool, while net solidification ensues along the trailing surface as the weld is produced. Filler metal may or may not be added. Fusion welding processes are typically more flexible with respect to possible joint geometries relative to friction welding and do not compromise the strength of the entire weld assembly.

3.3 Review of Studies on Joining of Al-MMCs

A review of joining of Al-MMCs reinforced with boron and graphite fibers by brazing, diffusion bonding, resistance welding and arc welding was given by Metzger in 1975 [3.21]. Low joint efficiencies were reported for all of these processes due to a lack of continuity of the fibers across the joint. Poor joint efficiencies were additionally blamed on damage to the fibers from contact with liquid Al during fusion welding or from deformation during solid state welding. Joining studies at the time were also plagued by the poor quality of the composites of that era. Similar results were reported in a 1978 review of joining of fiber reinforced MMCs by Sakamoto [3.22].

As mentioned earlier, interest in discontinuously (i.e. particle or whisker) reinforced aluminum (DRA) MMCs has recently grown due to their greater ease and lower cost of production as well as their relatively isotropic properties. A more recent review of joining of DRA-MMCs has been reported by the author of this dissertation [3.23]. Of the reinforcement materials used in DRA-MMCs SiC and Al₂O₃ are the most common. The remainder of this review focuses mainly on fusion welding of SiC DRA-MMCs. The following review is divided into sections on arc welding, capacitor discharge and flash welding, and high energy beam welding (laser and electron beam welding). The papers in each of the major sections are discussed in chronological order.
3.3.1 Arc Welding

A handful of papers have been published on arc welding of SiC DRA-MMCs [3.24-3.28]. Most of the studies employed the GTA or GMA welding processes. Several of the important articles are reviewed in this section.

Ahearn and Cooke

Ahearn and Cooke have reported on gas tungsten arc welding (GTAW) and gas metal arc welding (GMAW) of 6061Al-1.0 Mg-0.6 Si, all wt.%/SiC/20w produced using powder processing [3.24]. Preliminary efforts to weld this material were plagued by excessive amounts of porosity and delamination in the heat-affected-zone (HAZ). Mass spectroscopy analysis was performed to identify the source of the porosity. Pressure and sample mass were monitored during heating of small samples in vacuum, and the compositions of the gases evolved as a function of temperature were determined with a mass spectrometer. Molecular hydrogen was found to be the primary volatile constituent evolved during heating. The authors suggested that the source of hydrogen was a hydrate or hydroxide resulting from a reaction of moisture with the 6061 powder during primary processing. Degassing of samples for 48 hours at 500° C prior to welding was found to result in the elimination of porosity.

Note that transport of molecular hydrogen through the samples by diffusion is less likely than diffusion of monatomic hydrogen even though molecular hydrogen was found as the most prominent species in the mass spectrometer during degassing. Diffusion of hydrogen through the sample probably occurs as monatomic hydrogen and recombination to diatomic hydrogen takes place at the surface. The recombined diatomic hydrogen is then evolved and detected by the mass spectrometer.
GTAW was performed using 4043 (essentially Al-5.5 wt.% Si) filler metal, while GMAW was accomplished with 5356 (basically Al-5.0 wt. % Mg) filler metal. Optimum parameters for butt joints between 3.2 mm thick plates of 6061/SiC/20w were developed for each welding process. A very wide joint gap (~ 2.5 mm) was employed to overcome the problems associated with the highly viscous composite and to allow flow of the filler metal to the root of the weld. A nearly complete lack of mixing between the parent material and the filler metal can be noted from the photomicrographs presented.

After welding, the samples were examined using optical microscopy (OM). Results of this examination demonstrated that the high viscosity of the composite prevented any mixing of the reinforcement into the weld pool, but the 6061 alloy matrix was continuous with the filler metal from the fusion zone (FZ). No mention of Al$_4$C$_3$ formation was made, but none was evident from inspection of the photomicrographs.

Mechanical properties of the welds were evaluated using microhardness and tensile testing. The hardness increased from about 80 VHN in the base material to a peak of about 150-160 VHN in the HAZ and falls to about 60 VHN in the FZ. The authors attributed the large increase in HAZ hardness to the formation of precipitates resulting from the reaction between the Al alloy and the SiC (presumably precipitates of Si).

This interpretation is unlikely since this reaction has been shown to proceed at a negligibly slow rate in the solid state [2.23.2.61]. A more likely explanation involves the formation of a dispersion of Mg$_2$Si precipitates in the HAZ as a result of the weld thermal cycle. The 6061 matrix can be precipitation hardened by the formation of fine (< 1µm in size) Mg$_2$Si precipitates that inhibit dislocation motion. The temperature of the degassing heat treatments was clearly high enough to dissolve any precipitates in the parent material. The
authors did not give details of the cooling rates after degassing, but the condition of the parent metal prior to welding was likely similar to the solutioned condition. The temperatures experienced by the HAZ during welding were sufficient to allow development of fine Mg$_2$Si precipitates and provide the microhardness profile shown.

Due to the complete lack of mixing with the base material, the tensile properties reported for the welds were well below those of the base material and largely equivalent to those of welds in monolithic Al alloys. For example, the tensile strength of the GTA welds made with 4043 filler metal was 181 N/mm$^2$, while the tensile strength of the base material in the T6 condition was in excess of 415 N/mm$^2$. The elastic moduli of the welds were not reported, but they were probably much less than that of the base material since the fusion zone did not contain any reinforcement.

While sound, pore free welds were produced in this study, the poor joint efficiency and low modulus of these welds negate their use in engineering structures. The choice of Al-MMCs in structures is normally based on their improved mechanical properties and performance relative to monolithic Al alloys. The use of more expensive Al-MMCs in structures with mechanical properties equivalent to those produced from monolithic alloys is cost prohibitive and is not justified.

Gedeon, Rudd, Lane and Lo

Two studies of the effects of matrix composition, filler metal composition, volume fraction reinforcement and heat treat condition on the GMA weldability of several Al/SiCp MMCs have been completed by Gedeon et al. [3,25,3,26]. Cast MMCs with both A356 and A359 (Al-0.40. Mg-9.0 Si, all wt. %) matrices with 0, 10 and 20 volume fraction SiC were examined. Multi-pass welds were produced on one inch thick plates in the as-cast and T6
conditions at travel speeds ranging from 20 to 45 ipm using 4043 and 4047 filler metals. Welding conditions were developed by establishing boundaries or "windows" for acceptable weld bead profiles in current-voltage (I-V) space. The distribution of the SiC reinforcement and the amount of weld porosity were assessed for each composition/SiC volume fraction/heat treatment combination using OM. The mechanical properties of weld samples that passed radiographic inspection were evaluated by tensile testing according to the AWS D1.2-90 structural welding code for aluminum.

The SiC particles were claimed to be relatively well distributed throughout the fusion zone, and a region of high particle concentration along the fusion boundary was not observed. The authors reported no evidence of Al₄C₃ formation, although no extensive phase analysis was performed. All of the weldments produced with the optimized welding conditions exhibited yield and tensile strengths in excess of the AWS D1.2 requirement for monolithic A356. Unfortunately, no data on the yield or tensile strength of the unwelded parent material were provided to allow determination of joint efficiencies.

While the SiC particles were uniformly arranged on a gross scale, they were however distributed along the intercellular regions of relatively coarse α-Al cells and were thus not as homogeneously dispersed as the authors claimed. In addition, the more rapid weld travel rates (relative to the two previous studies) probably aided in limiting the formation of Al₄C₃. However, it is likely that some amount of Al₄C₃ did form (especially in the slower welds made on the plates with the lower Si content matrix with the lower Si filler metal) but was undetected in the examination with OM at 500x magnification.

The presence of porosity and a lack of fusion on the back side of the first weld pass were reported as the most common weld defects found in this study. The porosity tended to
concentrate near the top of each weld bead. Thus, grinding the top of the previous weld bead before proceeding with successive passes was found to limit porosity. The lack of fusion on the back side of the root pass presumably occurred due to an absence of shielding gas on the back side of the weld joint that allowed oxidation of the liquid Al matrix during welding and the formation of “cold laps” or “cold shuts”. Inert gas shielding of the back side of the joint during welding of the root pass may mitigate this problem.

**Thompson and Fenn**

Thompson and Fenn used GTAW to repair Al-MMC castings of 2124/SiC/20p [3.27]. Grooves were ground into the castings and filled to simulate repair welds. Initially, pressed “pills” of powdered Al alloy and SiC were pushed into the grooves and remelted. However, considerable porosity and lack of fusion resulted even when the size of the pills was reduced to 1 mm thick. Subsequently, loose powder mixes were used. Powder was poured into the groove and then remelted in a single pass. This process was repeated until the groove was filled. Somewhat greater success was achieved with the loose powder, however, problems with porosity, weld pool viscosity, poor surface finish and lack of fusion persisted. A range of welding parameters was examined, and $\text{Al}_4\text{C}_3$ formation was claimed to be limited by minimizing heat input; i.e. by decreasing current and increasing weld travel speed. However, the extent of defects due to lack of fusion between passes increased with decreasing heat input. The presence of $\text{Al}_4\text{C}_3$ was determined mainly by the use of optical microscopy.

The problems with porosity and lack of fusion likely derive from the presence of Al hydroxides on the powder surfaces. The authors should have considered cleaning of the powder immediately prior to welding. In addition, some $\text{Al}_4\text{C}_3$ was present in all of the welds produced, including those made at the lowest heat inputs. Formation of $\text{Al}_4\text{C}_3$ might have been limited if additions of Si additions had been made to the Al powder.
Hill, Wilkinson and Fenn

In a second paper by Fenn and co-workers, the authors employed consumable inserts machined from Al-MMCs to join tubes of 2124/SiC/20p using GTAW [3.28]. Consistent with the results of the previous paper, Hill et al. found that Al₄C₃ formation was lessened by minimizing the weld heat input. However, the results were not always repeatable, and problems with lack of fusion and viscosity were encountered as in the previous work.

3.3.2 Capacitor Discharge Welding and Flash Welding

Capacitor discharge welding (CDW) and flash welding (FW) share some common traits. Both processes can provide rapid thermal cycles and both involve displacement of much of the liquid metal from the weld pool. They differ largely in the amount of pressure applied to the joint during welding. A brief description of each process is given in Appendix A.

Devletian

The use of CDW for the joining of Al/SiC MMCs has been studied by Devletian [3.29]. The materials investigated included 6061/SiC/20p and 40p as well as 6061/SiC/48f. The Al/SiC MMCs were joined to themselves and to monolithic wrought 6061-T6 and 2024-T6 Al alloys. No mention was made of the methods of production of the Al/SiC MMCs. (They were most likely manufactured by powder processing.) Microstructures of the welded samples were characterized using OM.

In his review, Devletian references the following equation for the displacement reaction between Al and SiC from the work of Iseki et al.:

\[ 4 \text{Al}(l) + 3 \text{SiC}(s) \rightarrow \text{Al}_4\text{C}_3(s) + 3\text{Si}(s). \] (Eqn. 3.1)
This form of the equation implies that the Si forms as a separate pure product. Hence, the author apparently fails to recognize the dissolution of Si into the Al-Si alloy during this reaction as discussed in the paper by Iseki et al [2.58,2.59]. The equation should be written as previously discussed to reflect the dissolution of Si into the Al-Si alloy:

\[ 4 \text{Al}_{\text{Al-Si}} + 3 \text{SiC}(s) = \text{Al}_4\text{C}_3(s) + 3 \text{Si}_{\text{Al-Si}} \]  

(Eqn. 3.2)

where the underline denotes solution in the Al-Si alloy. Since this paper by Devletian was one of the first to discuss joining of Al/SiC MMCs, it has become one of the most referenced articles on the subject, and unfortunately, this oversight has been copied many times in subsequent papers by other authors.

CD welds were produced using cylindrical rods 0.125 to 0.25 inches in diameter. No details were given on the weld parameters used, only that parameters similar to those suitable for Al were employed. The fusion zones of the CD welds made in this study were found to be sound and almost indistinguishable from the parent material. Due to the expulsion of much of the liquid metal during welding, the fusion zones were generally very narrow (~100 μm). No porosity and no evidence of reaction between the SiC and Al alloy matrix were observed during inspection using OM.

The absence of porosity resulted from rapid thermal cycle and the displacement of the weld pool as well as from solidification of the narrow fusion zone under compression. The author attributed the lack of reaction products to a low degree of superheat and the rapid cooling rates inherent to the CDW process. However, expulsion of the hottest regions of the weld pool also play a large role in limiting the presence of reaction products in the final weld microstructure. Measurements showed that more than 50% of the weld pool was lost.
as spatter during welding. Reaction products that form during arcing are presumably carried away when the bulk of the weld pool is displaced and are not present in the final weld microstructure. Collection and examination of solidified drops of spatter for the presence of reaction products would have aided interpretation of the results. Moreover, the absence of reaction products can be attributed to the rapid nature of the entire thermal cycle especially the heating portion, not just the cooling portion of the cycle as the author claims.

Tensile testing of the weld joints was not performed, but some of the weld joints were destructively tested in tension. Failure of these weld joints occurred away from the fusion zone in the parent material indicating adequate joint strength.

Schwartz, Devletian, Chen and Gould

A paper by Schwartz et al. compared CDW and FW of 8009 (Al-8.5 Fe-1.3 V-1.7 Si, all wt.%)/SiC/l1p and 2009 (Al-3.8 Cu-1.3 Mg, all wt.%)/SiC/15w [3.30]. Both MMCs were produced by powder processing followed by extrusion to final size. CD welds were produced on cylindrical rods 0.6 cm in diameter using weld energies of 225, 324 and 486 joules. Flash welds were effected on plates 0.2 cm thick with an upset rate of 18 to 25 cm/sec using a DC power supply. Inert gas shielding was provided during FW to limit entrapment of oxides. The CD welds were examined with OM and TEM, while the flash welds were characterized using only OM. Tensile testing was performed on welds made with both processes.

The microstructure of the FZs of the CD welds was characterized by a nonhomogeneous distribution of reinforcement and a network of cracks. The fusion zone was about 100 μm in width. Although the authors did not mention this, the nonuniform distribution of SiC particles does not appear to result from pushing and entrapment during solidification. Heat
flow in CD welding is known to be one-dimensional in nature and mainly parallel to the long dimension of the rod [3.29]. Hence, solidification fronts are expected to grow inward from the parent material on both sides of the fusion zone, and if pushing is to occur, the SiC is expected to be aligned at the center of the fusion zone parallel to the bondline where the two solidification fronts meet. However, the SiC was nonuniformly distributed along to the bondline. The inhomogeneous distribution of SiC in the FZ is more likely a result of the retention of the banded distribution of reinforcement evident in the parent material.

The tensile joint efficiency of the CD welds produced with 324 and 486 joules was approximately 75% for room temperature testing, while that of the welds made with 225 joules was below 50%. Failure of the CDW tensile tests typically occurred through the FZ. The fracture surfaces for these specimens displayed brittle transgranular characteristics as well as large cracks for all weld energy levels. Electron diffraction studies identified the presence of tiny needles of Al₄C₃ (a few microns in length) in the FZ. The Al₄C₃ needles are too small to account for the extensive cracking and poor mechanical properties of these welds. In contrast to the previous paper, the authors claimed that superheating of the liquid gave rise to extreme temperatures in the FZ. This statement is especially curious since the author of the previous paper (Devletian) was one of the co-authors of this paper. The presence of Al₄C₃ was rationalized on the basis of these high temperatures. CD welds were not performed on the 2009/SiC/15w since the authors thought that the results would also be poor.

The forging action associated with the FW process resulted in a bending of the banded or striated base material of the 8009/SiC/1lp from a direction normal to the bondline to one parallel to the bondline. The deformed region extended more than 0.75 mm on both sides of the bondline. The fusion zone was approximately 40 µm wide, about half of that seen in the
CD welds. No evidence of Al₄C₃ or any reaction products was found during examination with OM. However, this does not necessarily imply the absence of Al₄C₃, since the Al₄C₃ found in the CD welds during TEM examination was not observed with OM. The tensile joint efficiency of the flash welds of 8009/SiC/11p was roughly 80% for room temperature testing.

Forging in the flash welds on 2009/SiC/15w also resulted in reorientation of the extruded texture and alignment of the long axis of the SiC fibers parallel to the bondline. The fusion zones of these welds were even narrower than those in the 8009/SiC/11p (< 20 μm), and the tensile joint efficiency was nearly 100%. The realignment of the SiC fibers did not appear to affect tensile properties. No discussion of the fracture surfaces of the flash welds was given.

3.3.3 High Energy Density Welding

High energy density welding (HEDW) is a term often used to describe a group of welding processes capable of producing power densities greater than 10⁶ W/cm². These processes include laser beam welding (LBW) and electron beam welding (EBW). The high power densities available with both processes allow rapid thermal cycles and the possibility for nonequilibrium processing. Surprisingly, a relatively large number of papers have been written on HEDW of SiC DRA-MMCs [3.31, 3.34-3.44, 3.47-3.49]. The most important of these papers are reviewed in this section.

Dahotre, McCay and McCay

A study of CO₂ LBW of A356/SiC/10p has been conducted by Dahotre et al [3.31]. Continuous wave (CW) CO₂ LB welds were produced without filler metal at a travel speed of 10 ipm using specific energies ranging from 5.3 x 10⁴ to 13.0 x 10⁴ J/cm². The relatively
slow travel speed used by these authors is somewhat curious. The high power densities available with LBW permit the use of very rapid travel speeds relative to arc welding processes which allow very rapid thermal cycles and the possibility for retention of SiC. By using such a slow travel speed, the authors do not fully exploit the capabilities of the laser.

Several questionable statements are made in the paper as discussed below. As in the work by Devletian, these authors referenced the work of Iseki et al., but did not recognize the dissolution of Si as a result of the reaction between the Al alloy and SiC. Furthermore, the SiC was said to actually “melt” during laser processing. The melting point of SiC is 2700° C [3.32]. While the temperature of the SiC may reach ~2000° C during LB welding, the SiC is more likely to dissolve into the liquid solution (see Figure 2.19) before reaching its melting point.

The FZ contained plate-like precipitates (identified as Al₄C₃ using X-ray diffraction) and blocky precipitates (apparently blocks of Si resulting from the dissolution of SiC) in a fine cellular matrix. The surrounding HAZ was reportedly comprised of the same plate-like precipitates, SiC particles and small blocks of Si. Actually the Al₄C₃ forms as curved needles, not plates as will be shown in subsequent discussions. There also appears to be some confusion with respect to the extent of the fusion zone. Strictly speaking, most of the weld region where the Al alloy matrix melts should be defined as a partially melted zone (PMZ) since the solid reinforcement locally persists during the process, but the use of the term fusion zone to indicate the region of melting of the Al alloy is not objectionable. By definition, the HAZ is a weld region where the heat of welding causes solid-state changes to the microstructure (i.e. no melting occurs). The quality of the optical micrographs in this paper is rather poor, but the Al alloy matrix in the regions referred to as the HAZ appears to have melted since some Al₄C₃ was present locally. The authors suggested that the Al₄C₃ formed
by solid-state reaction in the region they called the HAZ, but this is unlikely since the kinetics are extremely slow in the solid-state as discussed earlier [2.23, 2.61]. This second region is actually part of the fusion zone. Thus, there are at least two separate regions of the FZ: a region along the weld centerline in which all of the SiC was lost and entirely replaced by \( \text{Al}_4\text{C}_3 \), and a second region surrounding the first where partial loss of SiC occurs along with the formation of limited amounts of \( \text{Al}_4\text{C}_3 \) [3.34].

The length and width of the \( \text{Al}_4\text{C}_3 \) plates were shown to increase with increasing specific energy, and the volume fraction of remaining SiC decreased as the volume fraction of \( \text{Al}_4\text{C}_3 \) increased. The second result is consistent with the displacement reaction for \( \text{Al}_4\text{C}_3 \) formation and conservation of C. The authors also asserted that an optimum combination of \( \text{Al}_4\text{C}_3 \) and SiC reinforcement can be attained in the FZ through control of the specific energy. This statement is not justified since the acicular morphology and corrosion problems associated with \( \text{Al}_4\text{C}_3 \) always make its presence undesirable. Clearly, the desired result is the retention of largely unreacted SiC particles.

The partially dissolved and roughened surfaces of the surviving SiC were claimed to provide a stronger interface between the matrix and the SiC. The authors referred to the work of Ehrstrom and Kool [3.33] in which Al alloy/SiC MMCs produced by melt spinning and compaction of composite RS ribbons were compared to identical MMCs made by blending and compacting of Al and SiC powders. The results of the work by Ehrstrom and Kool suggested that strong bonding was not achieved between the matrix and SiC manufactured by the powder processing and that a strong bond can only be formed if the SiC is in contact with liquid Al for a short period of time. Since the SiC already contacted the liquid A356 matrix during production of the MMC, the contact with the superheated liquid matrix during LBW provided no further improvement to the strength of the bond. In fact, this contact
has been shown to result in serrating and faulting of the SiC [2.20, 2.22, 2.27, 2.28] which only serve to increase stress concentrations at the interface during loading and weaken the interface. While the serrated and roughened surfaces of the SiC may promote mechanical interlocking between the matrix and reinforcement, they do not improve mechanical properties.

Vickers microhardness traverses were performed from the centerline of the FZ to the parent material using a 300 gram load. The hardness consistently averaged approximately 140 VHN in a region termed the “laser melted region” (the inner FZ where no SiC remained) and fell through the region called the “HAZ” (outer FZ where some SiC remained) to an average value of about 60 VHN in the parent material. The authors ascribed the high hardness of the “laser melted region” to the existence of dislocations and precipitates along the dislocations, but failed to appreciate the contributions to hardness provided by the much finer grain size (relative to the parent material) and the presence of blocks of Si.

Cola, Lienert, Gould and Hurley

A similar investigation of LBW of 4.8 mm plates of A356/SiC/15p-F was undertaken by the author of this dissertation [3.34]. In contrast to the previous paper, the CW CO₂ LB welds produced in this study were made at travel speeds up to 230 ipm. Pulsed Nd:YAG LB welds were also completed using pulse lengths from 0.5 to 2.0 milliseconds. Hence, both of these processes yielded very rapid thermal cycles relative to the work of Dahotre et al. and afforded a greater possibility for retention of the SiC reinforcement during welding. Previous work by the author of this dissertation had shown that the rapid thermal cycles provided by pulsed Nd:YAG LB welding were successful in limiting dissolution and coarsening (during welding) of the fine intermetallic dispersoids responsible for strengthening a rapid solidification processed (RSP) Al-Fe-Mo alloy.
The CO₂ LB welds were made at various power levels and travel speeds to give linear heat inputs of 400 to 1000 J/in. The pulsed Nd:YAG LB welds were generated at 200 W (average power) at pulse frequencies from 10 to 40 Hz to yield pulse energies of 5 to 20 J. Travel speed for these welds were chosen to maintain a spot overlap of 65 to 70 %. After welding, metallographic mounts were prepared for microstructural characterization without contacting water to limit damage to the Al₄C₃. Unetched samples were examined with OM, and heavily etched samples (0.5 % HF) were inspected using an SEM with EDS capabilities.

The fusion zones of the CO₂ LB welds made in this study exhibited three distinct regions. The upper central region contained large amounts of needle-shaped phases alleged to be Al₄C₃ and Si-rich blocky phases claimed to be Si in the Al alloy matrix, with little remaining SiC. This region was spike shaped and encompassed less than 25 % of the weld cross-section. The Al₄C₃ needles in this region were not continuous with any SiC and were relatively long (up to ~50 μm). Furthermore, the needles were curved and were randomly oriented. These observations suggested that the Al₄C₃ in this region did not form by the previously discussed direct displacement reaction, but rather nucleated and grew from the liquid during cooling. The SiC was claimed to dissolve during LBW resulting in a hypereutectic solution of Si and C in liquid Al. The blocks of Si appeared to have grown on the Al₄C₃ needles in this region during hypereutectic solidification, and the matrix was believed to have solidified as primary α aluminum (~ 3μm cell spacing) with eutectic at the cell boundaries. The rapid solidification rates resulted in a very uniform distribution of the Al₄C₃ needles in the central region.

The central region of the CO₂ LB welds was surrounded by a very narrow region in which the SiC had partially reacted to form Al₄C₃ and Si. The partially reacted zone (PRZ)
did not appear to contain blocks of free Si, and solidification of the matrix in this region was alleged to have taken place as a hypoeutectic Al-Si alloy with a Si content somewhat higher than the nominal matrix composition. Thin needles of Al$_4$C$_3$ seemed to have grown from the SiC in the PRZ along distinct directions suggesting an orientation relation with the SiC. These needles were straight and were narrower and shorter than those seen in the central region. Surrounding the PRZ was a large region in which the SiC appeared unreacted. The unreacted zone (URZ) comprised ~ 70% of the total weld volume. The underlying matrix of the URZ exhibited a structure of a Al cells (~3 μm cell spacing) with intercellular eutectic. The rapid solidification rates promoted a very uniform distribution of the SiC reinforcement in the URZ. The SiC particles were typically dispersed along the cell boundaries indicating entrapment had occurred during solidification. Similar microstructural characteristics were also observed in the pulsed Nd:YAG LB welds.

The size and distribution of the Al$_4$C$_3$ in the central region of the CO$_2$ LB welds were observed to change as a function of distance from the top center of the weld. The needles of Al$_4$C$_3$ became finer and more widely spaced as the distance (depth) from the weld crown increased. Variations in the heat input of the CO$_2$ LB welds resulted in different weld penetration depths. As heat input was increased, the weld penetration increased, as did the depth of the central region containing Al$_4$C$_3$. Interestingly, the width of the central region of the CO$_2$ LB welds did not change with variations in heat input. The width of this region was shown to be approximately the same size as the focused beam diameter as calculated using nonlinear optics considerations. This observation suggested to the authors that the central region was associated with the high temperatures experienced near the keyhole.

The authors suggested that the nature of the three distinct regions of the FZs of the CO$_2$ LB welds was related to the stability of the SiC in the liquid Al alloy matrix throughout the
local transient thermal cycle. They proposed a qualitative model for microstructural development of the three regions. The central region interacted most closely with the laser beam and consequently experienced a thermal cycle sufficient to promote dissolution of the SiC into a liquid solution. Peak temperatures were thought to approach the vaporization temperature of Al (~2700° C). The dissolution of 15 volume % SiC in a matrix that already contained approximately 7 wt. % Si was claimed to result in a liquid solution of 15 at. % Si and 7.5 at. % C at peak temperature according to the ternary phase diagram [21]. On cooling, the Al₄C₃ was thought to precipitate from the liquid by consuming all of the carbon from solution. Upon further cooling below the liquidus temperature of the remaining hypoeutectic Al-Si solution, blocks of primary Si were indicated to precipitate and grow on the Al₄C₃. At the Al-Si eutectic temperature, the remaining liquid solidified invariantly as a eutectic. The structure of the matrix was described as α-Al cells with intercellular eutectic.

The material in the PRZ was further from the keyhole and experienced a somewhat lower peak temperature. The time at temperature was sufficient to partially dissolve some of the SiC particles and allow enrichment of the liquid solution with Si and C. Needles of Al₄C₃ were said to nucleate and grow on the surviving SiC particles upon cooling by depleting the solution of C. The remaining liquid, slightly enriched in Si, then solidified as a hypoeutectic Al-Si alloy.

The material in the URZ was furthest from the heat source and underwent the lowest peak temperature. The time at temperature endured by the SiC in the URZ was not sufficient to cause considerable dissolution on heating. Thus, no Al₄C₃ was formed on cooling. Subsequently, the composition of the Al alloy matrix was unchanged, and the matrix solidified as a hypoeutectic Al-Si alloy of the original composition with α-Al cells and intercellular eutectic. Similar microstructural results were reported by other workers in subsequent papers [3.35-3.37]
The major shortcomings of this work relate to the inabilities of the characterization techniques to positively identify the nature of the phases found in the weld microstructures. The Al₄C₃ was distinguished by matching of its needle-like morphology from micrographs published by previous workers who had also performed X-ray diffraction (XRD) [2.21, 2.58, 2.59, 3.24]. No attempts were made to distinguish Al₄C₃ from other possible reaction products such as 2Al₄C₃·SiC (Al₈SiC₇) and Al₄C₃·SiC (Al₄SiC₄). The EDS spectra taken from the blocky phase showed Si as the major component along with a smaller amount of Al. The blocky phase was identified as Si based on its light gray color and the EDS data, as well as from previous work [2.21]. The presence of Al in the spectra was assigned to Al from the matrix in the interaction volume of the electron beam during analysis.

Furthermore, the calculations of the composition of the liquid solution are not correct. Dissolution of 15 volume % SiC in a matrix of 7 wt. % Si does not give a solution of 15.0 at. % Si and 7.5 at.% C. This composition falls into the compatibility triangle for Al + Al₄C₃ + Al₄C₃·SiC and the primary crystallization field for 2Al₄C₃·SiC according to the phase diagrams presented in the previous chapter. This implies that the first solid to form on cooling should be 2Al₄C₃·SiC not Al₄C₃ and that the final microstructure should contain Al, Al₄C₃ and Al₄C₃·SiC. Furthermore, the description of microstructure of the matrix was consistent with that of a hypoeutectic alloy, but the matrix was claimed to solidify initially as a hypereutectic alloy followed by invariant solidification as a eutectic. The authors offer no explanations for these inconsistencies.

Dahotre, McCay, McCay and Gopinathan

In a series of very similar papers by Dahotre and coworkers, attempts were made to limit reaction of the SiC in 3 mm sheets of A356/SiC/10p and 20p MMCs using pulsed CO₂ LBW at 60 ipm with duty cycles ranging from 50 to 91% [3.38-3.41]. Dutycycle is de-
fined as the ratio of beam on time to the total time between pulses.) Microstructures of the weld samples were characterized using OM, TEM and EDS. Mechanical properties of weld samples were evaluated by tensile testing.

The parent material of the MMCs contained $\alpha$ and $\beta$ SiC in a hypoeutectic matrix of primary $\alpha$-Al cells and intercellular eutectic. The FZs of the LB welds were comprised of $\alpha$ SiC, $\text{Al}_4\text{C}_3$, and $\text{Al}_4\text{Si}_2\text{C}_5$. Despite the claims by the authors, tailoring of the weld thermal cycle via pulse processing does not eliminate loss or modification of the SiC particles. The $\alpha$ SiC exhibited a serrated interface with the matrix, and no $\beta$ SiC was found in the FZs. $\beta$ SiC is considered unstable above 2000°C, and the lack of $\beta$ SiC in the weld microstructures was offered as proof that the peak temperatures in the FZ exceeded this temperature. The presence of $\text{Al}_4\text{Si}_2\text{C}_5$ in the fusion zones is somewhat curious since its existence in the Al-Si-C system is not widely accepted. As in their first paper [3.31], the authors identified two distinct regions of the weld zone. The spatial variations in microstructure followed the same trends for all of the pulse duty cycles used. The shorter pulse duty cycles generated the least changes in the weld microstructure, while the longer duty cycles produced extensive modification of the SiC and formation of reaction products.

Several of the questionable statements made in their previous paper [3.31] were repeated by the authors in this paper. For example, they again claimed that the SiC melted during heating and that the serrated edges of the altered SiC contributed to improved mechanical properties. However, they did recognize melting of the matrix in the zone previously called the HAZ and correctly identified this zone as a PMZ in this paper.

Strangely, the weld travel speed was not changed with duty cycle to maintain a constant pulse overlap. The pulses of the shorter duty cycle welds were either separate (50%) or
barely overlapping (57\%). While the longer duty cycles (80 and 91\%) resulted in excessive overlap. Thus, both the number of melt/solidification events experienced at any location as well as the time and peak temperature of the thermal cycle varied according to duty cycle. Locations in the 50\% duty cycle welds underwent at most one melt/solidification cycle, and each pulse was placed at a previously unheated position. The extent of preheat (and thus the thermal cycle) experienced by each successive pulse and the number of possible melt/solidification events at any location increased with increasing duty cycle. Most of each pulse in the 91\% duty cycle welds was placed upon of a previously made pulse as it was still cooling, and any position may have experienced two or three melt/solidification cycles. A better comparison of the effects of duty cycle involves varying travel speed to maintain a constant pulse overlap and number of melt/solidification cycles.

The welds produced at the lowest duty cycles were not continuous and were not full penetration welds. The greatest yield and tensile strengths were realized from the weld samples produced with the intermediate duty cycles (67 and 74\%). The presence of large amounts of Al$_4$C$_3$ and modified SiC in the welds produced with the longest duty cycles (80 and 91\%) resulted in poor mechanical properties. The SiC particles and precipitates along the interface between the SiC and matrix were claimed to interact with dislocations and produce improved mechanical properties. This statement is misleading since the Al/SiC interface is incoherent, and the spacing of the SiC particles is too provide effective barriers for dislocations. However, the loops of mismatch dislocations generated due to the CTE mismatch between the matrix and SiC do interact with forest dislocations during deformation. As in their previous paper, the authors again fail to consider the effects of microstructural refinement (finer grain size and second phases) on the mechanical properties.
Meinert and Co-workers

Meinert et al. have proposed a novel method to preclude Al$_4$C$_3$ formation during CO$_2$ LB welding of Al/SiC composites by the use of Ti additions in the weld joint [3.42-3.43]. They offered plots of free energy of formation of SiC, Al$_4$C$_3$, TiC and ZrC as proof that Ti additions would limit Al$_4$C$_3$ formation. LB welds made with Ti filler additions to 6061/SiC/20w were claimed to contain no Al$_4$C$_3$. Instead, particles rich in Ti and C were found in a matrix containing Ti, Al and Si. Few remaining SiC particles were noted. The results of the paper were considered preliminary and efforts to determine the optimum level of Ti addition were suggested.

Meinert and Martukanitz

These authors continued the work described above in a third paper [3.44]. They suggested that the presence of Ti (or Zr) in the liquid solution will result in the formation of TiC (or ZrC) instead of Al$_4$C$_3$. Optimum levels of Ti (or Zr) were sought to suppress Al$_4$C$_3$ formation and limit development of brittle intermetallic phases. LB welds were performed on 2 mm thick sheets of 6061/SiC/20w at approximately 18 ipm with 1.4 kW of power using shims of various thickness pressed in the weld joint between the two plates to be joined. The phases present in the weld microstructures were characterized by OM, SEM and electron microprobe analysis. To allow comparison with experimental work, the authors also used the SOLGASMIX computer program in an attempt to model phase equilibria in the Al-Ti-Si-C system in the temperature range of 900 K to 2000 K. SOLGASMIX is a FORTRAN computer program that can determine chemical equilibrium in multi-component systems by minimization of the total system free energy [3.45, 3.46].

In the analysis section of this paper, the authors stated that data on the free energy of formation indicated that several metallic carbides are likely to supplant Al$_4$C$_3$. Based on the
plot of free energy of formation vs. temperature, the authors suggested that Ti and Zr show
the most promise in eliminating $\text{Al}_4\text{C}_3$ formation during welding of Al/SiC composites.

Determination of the equilibrium compositions at each temperature using SOLGASMIX
required knowledge of the activities of Ti (or Zr), Al and Si in solution. However, the avail­
able activity data were limited to the binary Al-Si and Ti-Al systems. Lack of activity data
for the Al-Zr system prevented the use of the SOLGASMIX program for analysis of Zr
additions. Activity data for liquid Al-Si solutions are readily available [2.16]. However,
activity data for liquid Al-Ti solutions have not been reported. The authors claimed that the
activity data for the (solid) $\beta$ phase from the Ti-rich end of the Al-Ti phase diagram was
used to approximate the activity of the liquid Al-Ti solution at the Al-rich end of the phase
diagram.

The use of Ti shims of various thickness resulted in the addition of 17, 26 and 34 wt. % Ti
to the fusion zones of the LB welds. Welds comprised of 17 wt. % Ti were found to
contain $\text{Al}_4\text{C}_3$, while those with 26 and 34 wt. % Ti contained TiC and nonstoichiometric
phases identified as $\text{Ti}_x\text{AlC}$, but no $\text{Al}_4\text{C}_3$. This result suggested that between 17 and 26 wt.
% Ti must be added to suppress $\text{Al}_4\text{C}_3$ production. However, the welds with 26 and 34 wt.
% Ti did contain phases identified as $\text{Al}_3\text{Ti}$ and $\text{TiSi}$. (Note that this phase identification is
only tentative since these phases were determined using electron microprobe and no dif­
fraction studies were performed.) Welds made with 23 wt. % Zr did exhibit $\text{Al}_4\text{C}_3$, while
those produced with 34 wt. % Zr did not. No mention was made of the presence of $\text{Al}_3\text{Zr}$ in
these welds.

SOLGASMIX analyses for the additions of Ti were performed for Ti levels ranging
from 0 to 30 wt. %. The results of these analyses revealed that approximately 22 wt. % Ti
was needed to assure that all of the SiC is converted to TiC and no C remained to form Al₄C₃. Note that this finding was consistent with the results of the experimental study which showed that between 17 and 26 wt. % Ti was needed to preclude Al₄C₃ formation. When the Ti content was below 22 wt. %, all of the Ti reacted with the SiC to form TiC, but excess SiC remained to react and produce Al₄C₃. For Ti levels above 22 wt. %, extra Ti was available to form Al₃Ti and TiSi. The results of the SOLGASMIX calculations also demonstrated that the temperature at which Al₄C₃ was initially produced increased as the Ti level was increased.

The experimental work described in this paper and the previous papers was effective in eliminating Al₄C₃ formation in the welds. Similar results were reported by other workers using Ti additions to the weld pool during laser welding of Al-MMCs [3.47, 3.48]. However, note that addition of Ti and Zr to the welds in Al/SiC MMCs is somewhat self-defeating in that it is expensive and adds considerable weight. Despite the apparent consistency between the thermodynamic analyses and the results of the experiments, the analytical work was seriously flawed. First, the plot of free energy of formation for Al₄C₃ was not normalized to a single mole of C reacted. The authors plotted the free energy for the reaction:

\[ 4 \text{Al}(s) + 3 \text{C}(s) = \text{Al}_4\text{C}_3 \]  \hspace{1cm} \text{(Eqn. 3.3)}

instead of the reaction:

\[ \frac{804}{3} \text{Al}(s) + \text{C}(s) = \frac{1}{3} \text{Al}_4\text{C}_3. \]  \hspace{1cm} \text{(Eqn. 3.4)}

This error results in an apparent shift in the free energy curve for Al₄C₃ formation to much larger negative values and does not allow a proper comparison of the differences in standard free energy of formation for the various carbides.
The authors claimed TiC and ZrC would displace Al₄C₃ base solely on the relative positions of the curves of the standard free energy formation. Even when the standard free energy for Al₄C₃ formation is correctly plotted, direct comparison of this curve with the curves for the other carbides is not alone sufficient for determination of the most stable carbide for the case of LB welding of Al-MMCs with Ti and Zr additions. Curves for the standard free energy of formation are only applicable to situations when all of the components are in their standard state; i.e. when they are in a pure state. LB welding of SiC reinforced Al-MMCs with Ti and Zr additions results in a liquid solution containing Al, Si, C and Ti (or Zr) as well as other minor elements, and solution thermodynamics must be employed to determine the most stable carbide.

Modeling of the reactions using the SOLGASMIX program then requires knowledge of the activities of all of the elements in the liquid solution. While it is recognized that no activity data exists for such a complicated multi-component system and that approximations using activity data to the Al-Ti and Al-Si systems are acceptable for the calculations attempted by the authors, extension or extrapolation of activity data for solid Ti-Al solutions (e.g. the β phase from the Ti-rich end of the Al-Ti phase diagram) as attempted by the authors is clearly not justified for Al-rich Al-Ti liquid solutions. Use of this activity data for the case at hand raises serious doubts about the accuracy of the results. The apparent matching between the analyses and the experimental results appears to result more from serendipity than accuracy of the method.

Ahearn and Cooke

Another study by Ahearn and Cooke involved electron beam welding (EBW) of 0.125 inch plates of 6061/SiC/20f [122]. This MMC was produced by powder processing and was degassed using the same procedure as that described previously [3.24]. EB welds were
made without filler metal using ~ 1kW of power at 15 ipm in a vacuum of 10^{-4} torr. Again, the use of a high power density process like EBW at 15 ipm is curious. The authors fail to appreciate the utility of EBW. Weld microstructures were characterized using OM and XRD, while the mechanical properties of the welds were evaluated by microhardness testing.

No evidence of remaining SiC was found in the FZ during inspection of the weld samples with OM, but a needle-like phase was observed. Results of XRD studies of material taken from the FZ showed that the SiC peaks were very small compared to the parent material and that the peaks for Al_{4}C_{3} and Si were very large. These findings suggested that the reaction between the SiC and the 6061 matrix went to completion. This result is consistent with the relatively slow travel speed used to produce the welds. The authors proposed that the high temperatures around the keyhole enhanced the reaction and precluded the use of EBW for joining Al/SiC MMCs. The porosity observed in the region described as the HAZ was claimed to have been caused by residual hydrogen bearing species remaining after degassing.

However, the pores were located away from any free surface and were too big to have developed during the relatively short thermal cycle of the EB weld. This region was likely not the HAZ but a region where the matrix had at least partially melted and resolidified without reaction of the SiC (like the unreacted zone or URZ as discussed in [3.34]).

Vickers microhardness traverses revealed a relatively high hardness (up to 280 VHN) in the FZ falling through the URZ to about 110 VHN in the parent metal. Note that as in the previous paper by these authors [97], the plates were not reheated to a T6 condition after degassing and were in a condition similar to the solutioned condition. The relatively high hardness of the FZ was attributed to some aspect of the Al-SiC reaction.
This microhardness data is somewhat hard to interpret. Microhardness indents were apparently made at a spacing of 0.5 mm, but the region described as the FZ was only 1 mm wide. Only three indents were associated with the FZ. One indent was placed in the center of the FZ, and one at each of the two boundaries between the so-called FZ and HAZ. Two of these three indents gave hardnesses of about 200 VHN, but the third (on one boundary) gave a much higher value of 280 VHN. Thus, it is not clear if this third data point is representative of the actual hardness of the FZ. Closer spacing of the indents and/or repeated traverses at different locations would have provided more information and allowed clearer interpretation. The authors are not definitive in their explanation of the high FZ hardness, although it likely stems from the presence of large volume fractions of Si and Al₄C₃ (all of the 20 vol. % SiC was reacted).

3.4 Summary

To recapitulate, the different fusion welding processes for welding of SiC DRA-MMCs have been employed with varying degrees of success. Considerable work must be done to improve the understanding of fusion welding of SiC DRA-MMCs if they are to be exploited as engineering materials. However, several general conclusions may be drawn from this review. The SiC DRA-MMCs with casting alloy matrices such as A356 tend to suffer fewer problems with hydrogen porosity relative to those MMCs produced by consolidation of wrought alloy powders, such as 6061, and SiC. Moreover, the higher Si content of the casting alloy matrices aids in limiting reaction and dissolution of the SiC. However, the casting matrices are less desirable since they have lower strength than the wrought alloys, and the presence of Si-rich phases along the cell boundaries tends to lower their toughness. SiC DRA-MMCs prepared by powder processing may be degassed prior to welding, but the high temperatures required for degassing tend to lower the mechanical properties of the matrix alloy as a result of recovery and recrystallization (for strain hardened matrices) or
coarsening and dissolution of strengthening precipitates (for precipitation strengthened matrices). Recovery of the mechanical properties of the precipitation strengthened matrices after degassing is possible via heat treatment, but this adds further cost and time to manufacturing.

Arc welding of plates of SiC DRA-MMCs typically requires joint preparation by machining of some type of V-groove. Hence, arc welding of these plates demands the use of filler material to replace the material removed by machining. Subsequently, the use of unreinforced filler metal results in the presence of less reinforcement and lower local mechanical properties (lower elastic modulus and tensile strength) in the weld FZ. The production of composite weld joints requires the development of proper composite filler materials. Nonetheless, the slow solidification rates inherent to the arc welding processes result in less than optimal distributions of the reinforcement and relatively large grain size in the FZ.

Welding processes capable of rapid solidification, such as CDW, FW, and LBW, offer the advantages of a more uniform distribution of the reinforcement and finer grain size in the FZ of SiC DRA-MMCs. Moreover, these processes do not require filler metal and are capable of producing true composite weld joints. CDW and FW also involve displacement of the hottest material from the weld pool including any undesirable reaction products. EBW has been shown to be ineffective for welding of SiC DRA-MMCs due to formation of $\text{Al}_4\text{C}_3$ and considerable amounts of porosity. However, the very slow travel speeds used do not allow rapid thermal cycles. and further work with EBW at rapid travel speeds may prove fruitful. Despite all attempts to tailor the thermal cycle of LB welds via control of specific energy and beam pulsing, LBW appears incapable of producing weld joints free of undesirable phases such as $\text{Al}_4\text{C}_3$ and ternary carbides in the central region due to direct
interaction with the beam. The rapid thermal cycles associated with LBW does however limit $\text{Al}_4\text{C}_3$ formation throughout the rest of the FZ. Clearly, further work needs to be done to improve our abilities to produce welds in SiC DRA-MMCs with properties approaching those of the parent material.
CHAPTER 4

OBJECTIVES

The utility of Al-MMCs was discussed in the first part of Chapter 2. Al-MMCs show considerable promise as structural materials, however their widespread use awaits further development of enabling technologies such as joining. The remainder of the reviews given in Chapters 2 and 3 focused on the problems associated with joining of SiC-reinforced Al-MMCs. The major problem associated with fusion welding of these materials appears to be related to Al₄C₃ formation in the weld zone. If SiC-reinforced Al-MMCs are to reach their full potential, then joining techniques must be found that eliminate Al₄C₃ formation. Additionally, joining processes/parameters that provide for refined microstructures and uniform reinforcement distribution should be developed.

Results of preliminary work by the dissertation author using LB welding at very rapid travel speeds showed significant potential in eliminating Al₄C₃ formation in much of the weld zone. Unfortunately large amounts of Al₄C₃ were found along the weld centerline of the LB welds. A need exists to understand how and why the microstructures evolve in the different regions of the LB welds so that steps can be taken to limit or completely eliminate Al₄C₃ formation. This task also requires accurate characterization to understand the microstructure.

Work by other authors has shown that EB welding of Al-MMCs is not successful in eliminating Al₄C₃ formation. However, these authors used very slow travel speeds with
EBW, and did not take full advantage of the process. EBW and LBW share certain traits in that both are capable of producing very rapid thermal cycles. Hence, EBW of may also enjoy success in limiting Al₄C₃ formation and should be investigated.

The ultimate goal in tailoring microstructures of a given material is to produce the best combinations of properties for a given application. Since Al-MMCs are targeted for structural applications where joining is required, there is a need to develop a database of mechanical properties for laser and electron beam welded Al-MMCs. Very little work has been done to measure tensile properties of welded Al-MMCs. The little work performed to date has been inconclusive and confusing. The conditions used did not allow an accurate comparison of properties. Specifically, some welds were actually intermittent, while others allow for repeated thermal cycling. The authors reported that the laser welds had properties that were superior to those of the base material despite the presence of large amounts of Al₄C₃. They did not recognize the effects of the disparity in grain size and other microstructural features on the mechanical properties. A proper study of mechanical properties that accounts for this disparity is required.

The objectives of this dissertation are aimed at fulfilling the needs outlined here. The specific objectives of this work are:

1) Produce sound EB and LB welds on a SiC-reinforced Al-MMC and determine the effects of power and travel speed on Al₄C₃ formation and the development of other microstructural features.

2) Characterize the microstructures of the welds with a view toward understanding their microstructural evolution.

3) Develop qualitative models for microstructural evolution of the EB welds and different
regions of the LB welds using mass balance calculations and the knowledge gained from the characterization phase.

4) Determine the cause for the large differences in microstructures between the EB and LB welds.

5) Attempt to measure mechanical properties of the LB and EB welds to discern if the differences in microstructure greatly affect properties.

The purpose of work described in the remainder of this document is to fulfill these objectives.
CHAPTER 5

EXPERIMENTAL PROCEDURES

Objectives of this work included producing sound welds on Al/SiC-MMCs, understanding the microstructural evolution in these welds and determining mechanical properties of these welds. The experiments aimed at completing these objectives are outlined in this chapter. Compositions, preparation and testing of the different Al-MMCs are described along with details of the various welding experiments. In addition, information concerning microstructural characterization is presented.

5.1 Materials

Three different Al-MMCs were used in the study: A356/SiC/15p, 359/SiC/10p and 359/SiC/20p. Nominal compositions for the three materials are given in Table 5.1 [5.1]. The matrices for all of the Al-MMCs were Al-Si-Mg casting alloys of the 35X family of alloys. The nominal A356 composition is Al + 6.5 to 7.5 wt. % Si + 0.25 to 0.45 wt.% Mg plus trace elements, while the nominal 359 matrix composition has more Si (8.5 to 9.5 wt. %) and more Mg (0.45 to 0.65 wt.%) in addition to the trace elements. These alloy matrices can be strengthened via precipitation of a distribution of $\beta''$ Mg Si. Alpha ($\alpha$)-SiC particles were used as the reinforcing phase for each Al-MMC, and all of the composites were manufactured by Duralcan USA. Note the different volume fractions of SiC for the three Al-MMCs as well as the slightly larger SiC particle size for the 359 base MMCs.
<table>
<thead>
<tr>
<th>Material</th>
<th>Wt % Al*</th>
<th>Wt % Si</th>
<th>Wt % Mg</th>
<th>Wt % Fe</th>
<th>Vol % SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>A356/SiC/15p</td>
<td>Rem.</td>
<td>6.5 - 7.5</td>
<td>0.25 - 0.45</td>
<td>0.2 max</td>
<td>15</td>
</tr>
<tr>
<td>359/SiC/10p</td>
<td>Rem.</td>
<td>8.5 - 9.5</td>
<td>0.45 - 0.65</td>
<td>0.2 max</td>
<td>10</td>
</tr>
<tr>
<td>359/SiC/20p</td>
<td>Rem.</td>
<td>8.5 - 9.5</td>
<td>0.45 - 0.65</td>
<td>0.2 max</td>
<td>20</td>
</tr>
</tbody>
</table>

* Plus trace amounts of Cu, Mn, Ti and Zr totaling < 1.0 %

Table 5.1: Material Compositions

The A356/SiC/15p MMC was consumed during microstructural characterization phase of this work and was used exclusively for that phase. The A356/SiC/15 was supplied in the form of an as-cast cylinder approximately two inches in diameter. The 359/SiC/10p and 359/SiC/20p MMCs were used mainly for production of mechanical test specimens. The 359/SiC/20p MMC was supplied in the form of an ingot about 30 inches in length and four inches deep. Melts of 359/SiC/10p were produced by diluting melts of 359/SiC/20p with proper combinations of master alloys. The 359/SiC/10p and 359/SiC/20p were cast into plates used subsequently to produce mechanical specimens, as described below.

5.2 Casting

To limit chemical reactions and casting porosity, the Al-MMCs (and master alloys) were charged into a SiO₂ crucible coated with BN and induction melted in a sealed quartz system. Prior to heating, the system was evacuated to a partial vacuum equivalent to -50 mm Hg. Heating and melting were carried out under desiccated HP argon gas at a positive pressure of +20 mm Hg. The melts were thus shielded until the system was opened for pouring. Peak pouring temperatures ranged from 715° C to 805° C for the 10 heats of
material produced. To achieve a rapid cooling rate and ensure a relatively fine grain size, small plates 3” x 4” x 3/8” in size were cast against a 7” x 4” x 1/2” Cu chill plate into a 1/16” thick stainless steel book mold. After casting, the plates were machined to 0.20” thick using polycrystalline diamond (PCD) tooling. To determine the presence of porosity and cold-shuts, machined plates were subsequently examined by ultrasonic C-Scan testing using a 5 MHz focused transducer (1/2” diameter). Subscale tensile samples three inches long with a one inch gage length were machined from the plates and tested at room temperature at a strain rate of $10^{-3}$.

5.3 Welding

Three different welding processes were utilized in this work: continuous wave (CW) CO$_2$ laser beam welding (LBW), conventional vacuum electron beam welding (EBW) and non-vacuum electron beam welding (NVEBW). All of the welds produced in this study were autogenous (i.e. no filler metals were used), bead-on-plate (i.e. no joint was used) welds. Samples were rigidly fixtured during welding and were allowed to cool after each weld.

5.3.1 Laser Beam Welding (LBW)

The parameters used for continuous wave LBW are collated in Table 5.2. The first two LB welds were produced on 0.2 inch thick plates of A356/SiC/15p at 200 ipm at sharp focus of a 12.7 cm focal length lens. These LB welds were protected from contamination by high purity helium gas flowing at a rate of 19 lpm through a 0.25 inch diameter nozzle. The theoretical beam diameter of the laser at sharp focus was calculated using nonlinear optic considerations at about 0.02 inches [5.2]. CW CO$_2$ LB welds were also completed on 0.05 thick sheets of A356/SiC/15p at sharp focus of a five inch focal length lens at 150 ipm travel.
speed using various powers. Helium was again used to shield the welds. This set of welds was used primarily to produce TEM thin foils.

<table>
<thead>
<tr>
<th>Weld ID</th>
<th>P (kW)</th>
<th>Speed (ipm)</th>
<th>Focus</th>
<th>Plate t. (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LBW-1</td>
<td>3.0</td>
<td>200</td>
<td>Surface</td>
<td>0.2</td>
</tr>
<tr>
<td>LBW-2</td>
<td>1.5</td>
<td>200</td>
<td>Surface</td>
<td>0.2</td>
</tr>
<tr>
<td>LBW-3</td>
<td>0.8</td>
<td>150</td>
<td>Surface</td>
<td>0.05</td>
</tr>
<tr>
<td>LBW-4</td>
<td>0.9</td>
<td>150</td>
<td>Surface</td>
<td>0.05</td>
</tr>
<tr>
<td>LBW-5</td>
<td>1.0</td>
<td>150</td>
<td>Surface</td>
<td>0.05</td>
</tr>
<tr>
<td>LBW-6</td>
<td>1.1</td>
<td>150</td>
<td>Surface</td>
<td>0.05</td>
</tr>
<tr>
<td>LBW-7</td>
<td>1.2</td>
<td>150</td>
<td>Surface</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 5.2: Laser Beam Welding Parameters

5.3.2 Electron Beam Welding (EBW)

Bead-on-plate electron beam (EB) welds were also produced on similar 0.2 inch thick plates of the A356/SiC/15p MMC with a 15 kW EB welding machine using a gun-to-work distance of approximately 11 inches in a vacuum of $10^{-4}$ Torr. The diameter of the electron beam at sharp focus was estimated at approximately 0.02 inches. EB welding parameters used are given in Table 5.3. Note that the power, travel speed and focus conditions of the first two EB welds are identical to those of the first two LB welds. The last two EB welds were made to study the effects of travel speed and focus condition on microstructural development.
5.3.3 Non-Vacuum Electron Beam Welding (NVEBW)

EB welds were also produced on similar 0.2 inch thick plates with a non-vacuum EB welding machine with a gun-to-work distance of about 3/8 inches. The total pressure around the weld area was approximately one atmosphere, and the diameter of the electron beam at the exit orifice was 0.01 inches. However, the beam diverged rapidly with distance from the exit orifice. The parameters used for NVEB welding are presented in Table 5.4. Again, note that the power and travel speed of the first two NVEB welds are identical to those of the first two EB and LB welds.

<table>
<thead>
<tr>
<th>Weld ID</th>
<th>I (mA)</th>
<th>V (kV)</th>
<th>Speed (ipm)</th>
<th>Plate t. (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NVEBW-1</td>
<td>5</td>
<td>150</td>
<td>200</td>
<td>0.2</td>
</tr>
<tr>
<td>NVEBW-2</td>
<td>10</td>
<td>150</td>
<td>200</td>
<td>0.2</td>
</tr>
<tr>
<td>NVEBW-3</td>
<td>20</td>
<td>150</td>
<td>200</td>
<td>0.2</td>
</tr>
</tbody>
</table>

Table 5.4: Non-Vacuum Electron Beam Welding Parameters
5.4 Microstructural Characterization

Characterization of the microstructure was undertaken on several size scales with a view toward understanding microstructural evolution in the LB and EB welds. Characterization was completed on samples from the welds and base material of the A356/SiC/15p using the following techniques: optical microscopy (OM)/image analysis, scanning electron microscopy (SEM) with x-ray energy dispersive spectroscopy (EDS), x-ray diffraction (XRD) and transmission electron microscopy (TEM) with EDS. In particular, the identity of the phases present in the welds and base material were determined by XRD and TEM diffraction work, and the Si content and the volume fractions of SiC and Al$_4$C$_3$ were tracked using SEM/EDS and optical image analysis.

5.4.1 Optical Microscopy

Optical microscopy allows for a lower magnification survey of microstructures. Transverse and planar longitudinal sections were removed from the welds and prepared using standard metallographic practices through 1 μm diamond paste followed by final polishing with a colloidal silica suspension. Exposure to water during preparation was limited as much as possible during grinding and polishing, and alcohol was used for rinsing between preparation steps to avoid dissolution of the Al$_4$C$_3$. The samples were not etched. Similar procedures were followed with samples from the base material of the A356/SiC/15p.

The welds were examined at magnifications up to 1000X using an optical microscope. Volume fractions of the various phases in the welds and base material were measured with a computer-based optical image analyzer interfaced with the optical microscope. The computer assigned one of 256 gray levels to each pixel of the image, and volume fractions of each phase were identified by setting upper and lower bounds on gray level spectra corre-
sponding to each phase. The computer determined the volume fractions for each phase by summing the fraction of pixels within the preset upper and lower bounds set for each.

5.4.2 Scanning Electron Microscopy (SEM)

SEM permits observation of microstructures at higher magnification, while EDS enables approximation of sample compositions. Selected samples of the EB welds were coated with ~100 Å of carbon and examined with a Phillips XL-30 (SEM) equipped with an EDS system and operated at 15 kV. Compositions of different regions were determined from EDS spectra obtained by rastering the electron beam over an area approximately 20 μm x 20 μm in size. Reported compositions were determined from standardless quantification of the EDS data. The estimated statistical error associated with this technique was ±0.5 atomic percent.

5.4.3 X-Ray Diffraction (XRD)

XRD allows determination of the crystal structures of the major phases present in a bulk sample. Small samples from the base material and LB welds of the A356/SiC/15p were examined in a Scintag PAD-V x-ray diffractometer using Cu Kα radiation and a 0.030° step increment. Note that the LB welds were rather small, and that samples from the LB welds contained about 50 percent base material by volume. No XRD work was done on the EB welds. The presence of a given phase was determined by matching the position of x-ray peaks with known lattice spacings.

5.4.4 Transmission Electron Microscopy (TEM)

TEM permits identification of individual phases in small samples via determination of composition using EDS and crystal structure utilizing electron diffraction. Disks 0.120 inches
in diameter were removed from the LB welds. EB welds and base material of the A356/SiC/15p. Electron transparent thin foils were produced by ion milling ground and dimpled disks to perforation using a Gatan dual ion mill or a Gatan precision ion polishing system (PIPS). Foils were examined with a Phillips CM-200 TEM/AEM operated at 200 kV. Several diffraction and imaging techniques were employed to determine crystallographic information for the various phases found. Diffraction patterns from the various phases were obtained in selected area diffraction and microdiffraction modes. Bright field (BF) and centered aperture dark field (DF) images were also recorded to determine the phase distributions in the base material and welds. Tilting experiments were also conducted to determine orientation relations. Compositional analyses of the microstructural constituents were performed using an EDS system with a thin Be window. As with the SEM/EDS data, compositions reported for the TEM/EDS were derived from standardless quantification of spectra and have a statistical error of about 0.5 atomic percent.

Analysis of diffraction patterns was aided by the use of the Desktop Microscopist program [5.3]. The program requires the input of space group and atom positions for the material being analyzed, and can provide simulated diffraction patterns. Kikuchi maps and other data useful for comparison with experimental results.

5.5 Summary

In this chapter, the experiments devised to fulfill the project objectives have been discussed. The compositions and production of the different Al-MMCs have been outlined as well as details of the various welding experiments. In addition, information concerning microstructural characterization has been presented. Results of the microstructural characterization studies are presented in the following chapter.
CHAPTER 6

MICROSTRUCTURAL CHARACTERIZATION

Results of the microstructural characterization of the base material as well as the LB and EB welds on the cast A356/SiC/15p MMC are reported in this chapter. Characterization of the welds involved the fusion zone only. No characterization of the weld heat-affected-zone (HAZ) was undertaken. The solidification cell size of the welds was found to be much smaller than that of the base material owing to the higher solidification rates of the welds. In general, the distribution of SiC in the welds was much more uniform than in the base material due to the rapid cooling rates of the welds. Additionally, the microstructure of the LB welds were found to differ considerably from the EB welds. On average, the weld cross-section areas for the LB welds were approximately 25% smaller than that for EB welds made at identical powers, travel speeds and focus conditions. Furthermore, the LB welds were somewhat wider than the comparable EB welds. The LB welds were found to contain much more of the undesirable $\text{Al}_4\text{C}_3$ phase relative to the EB welds. The fusion zone of the LB welds contained three microstructurally distinct regions, while the fusion zone of the EB welds was microstructurally uniform. The microstructural information is summarized in a table at the end of this chapter. Knowledge of the microstructures of the EB and LB welds are used along with rationale developed in the next two chapters to explain microstructural evolution in the welds.
6.1 Base Material

6.1.1 Optical Microscopy and Scanning Electron Microscopy

Optical micrographs of the as-received microstructure of the cast A356/SiC/15p MMC are given in Figures 6.1a and b. The apparent cell spacing of the matrix alloy of approximately 75 µm indicates a cooling rate of 1.5 to 2 °C s⁻¹ during casting [2.8]. Needles of Al-Si eutectic were distributed along cell boundaries. The Si content of the A356 alloy matrix was determined by quantifying EDS spectra in the SEM from regions comprised of several cells containing no SiC particles. A Si content of 7.0 ± 0.5 % by weight was found using a standardless EDS analysis in the SEM. The SiC particles were roughly 15 to 20 µm in size and were often clustered at intercellular regions as a result of pushing and entrapment during solidification as discussed in Chapter 2. A volume fraction of SiC of 17.3% with a standard deviation of 6.3% was determined with the computer assisted image analysis of several optical micrographs at 200x and 400x magnification.

6.1.2 X-Ray Diffraction

X-ray diffraction (XRD) allows determination of the crystal structures of the major phases present in bulk samples. Figure 6.2 is a plot an x-ray diffraction spectrum from a base material sample. The plot gives intensity as a function of the diffraction angle, θ. The spectrum covers diffraction angles from 10° to 55°. All of the peaks were indexed and found to correspond to α-Al, Si and 6H-SiC.

6.1.3 Transmission Electron Microscopy

Transmission electron microscopy (TEM) permits identification of individual phases in small samples via determination of composition using EDS and crystal structure utilizing
Figure 6.1 (a) and (b): Optical micrographs of the microstructure of the A356/SiC/15p base material. Gray arrows in (b) indicate Al-Si eutectic phases, while black arrows highlight clusters of SiC along cell boundaries of primary α aluminum.
Figure 6.2: X-ray diffraction spectrum for A356/SiC/15p base material. Spectrum acquired using Cu K$_\alpha$ radiation. Major peaks were found to correspond to Al, Si and 6H-SiC.
electron diffraction. TEM also allows imaging of microstructural features such as grains, precipitates and dislocations. The TEM results confirmed the XRD results. Figure 6.3 a is a two-beam bright field image of the α-Al matrix recorded using the g=111 diffraction vector. The two-beam diffraction condition (s>0) is shown in the inset of Figure 6.3 a. Tangles of dislocations with b=1/2 <110> are clearly visible. Figure 6.3 b is a centered-aperture dark field image of the α-Al matrix taken with the g=022 diffraction vector. Again, dislocations with b=1/2 <110> are visible. Figure 6.3 c is an EDS spectrum from the α-Al matrix. Consistent with the SEM EDS results, the matrix was rich in Al and Si.

Figures 6.4 a and b are CBED and SAD patterns of the [111] zone axis of the α-Al matrix, respectively. Consistent with the [111] zone axis of the α-Al matrix, the CBED pattern displays three-fold symmetry. As expected, the SAD patterns of the α-Al matrix were indexed consistently with an fcc crystal structure (Fm3m. JCPDS file #4-787) with a lattice parameter of 4.0497 Å [6.1]. Figures 6.4 c, d and e are two-beam bright-field images recorded using different 022 type diffracting vectors from the [111] zone axis shown in Figure 6.4 b. The precipitates indicated by the double-headed arrows appear to be the β'' strengthening precipitates. Projections of the three variants of the β'' precipitates can be seen in the figures. Similarly, Figure 6.4 d shows projections of two of the β'' variants recorded using the g=111 diffracting vector of the α-Al matrix.

As discussed above, the base material also contained Si. Figure 6.5 a is an SAD pattern of Si. Reflections from both the zero and first order Laue zones are visible. This SAD pattern was indexed consistently with the [011] zone axis of diamond-cubic Si (Fd3m. JCPDS file# 27-1042) with a lattice parameter of 5.431 Å [6.2]. The Si also exhibited some type of faulting. Figure 6.5 b is two-beam bright field image of Si recorded using the g=111 diffracting vector for Si near the [011] zone axis. Further work is needed to determine the
Figure 6.3: (a) TEM bright field image of the alpha-Al matrix of the base material. Inset shows the two-beam condition $g=111$ Al. $a/2<110>$ dislocations are visible. (b) TEM dark field image of the alpha-Al matrix ($g=022$) Al. $a/2<110>$ dislocations are visible. (c) EDS spectra from alpha-Al matrix of the base material. The matrix is rich in Al and Si.
Figure 6.4: (a) Convergent beam electron diffraction (CBED) pattern from the [111] zone axis of the $\alpha$-Al matrix. (b) Selected area diffraction (SAD) pattern from the [111] zone axis of the $\alpha$-Al matrix. (c), (d) and (e) Two-beam bright field images recorded near the [111] zone axis of the $\alpha$-Al matrix with different diffracting vectors. The 3 different variants of the $\beta''$ strengthening precipitates are indicated by the two-headed arrows. (f) A two-beam bright field image recorded near the [110] zone axis of the $\alpha$-Al matrix with the $g = 111$ diffracting vector. Only two of the variants of $\beta''$ strengthening precipitates are visible.
Figure 6.5: (a) [011] zone axis of Si. (b) Two-beam bright field image of Si recorded near the [011] zone axis using $g = 1 \overline{1} T$. Fringes apparently stem from faulting. (c) Multi-beam bright field image of twinned Si. (d) EDS spectrum from Si.
exact nature of the faulting. A second type of faulting was also found in the Si grain shown in Figure 6.5 c. This Si grain was situated between a SiC particle and a grain of the α-Al matrix. The faulting appears to stem from the formation of twins. Stresses generated by differences in CTE during cooling of the base material after casting may be the cause of the faulting. Figure 6.5 d is an EDS spectra of the Si phase shown in Figure 6.5 b. Note that the Al peak arises from excitation of the surrounding Al.

The presence of Si was unambiguously determined by performing tilting experiments to identify three zone axes. Figures 6.6 a, b and c are SAD patterns that were consistently indexed as the [001], [013] and [114] zone axes of Si. Measured angles between the three axes corresponded very closely with published and calculated angles providing further evidence. Superlattice-type reflections can be seen near the 200 type reflections in the [001] and [013] patterns. These reflections are thought to arise due to the faulting shown previously. Further diffraction studies are required to determine their exact nature. Figure 6.6 d is a section of a Kikuchi map near the [001] zone axis of Si that shows the spatial relation between the three zone axes and the Kikuchi bands followed during tilting between zone axes.

Most of the SiC particles in the base material were found to display the 6H polytype, although particles of the 4H polytype were also encountered. SAD patterns of the 6H-SiC were indexed consistently with a hexagonal crystal structure (P6/mc, JCPDS file #29-1131) with a = 3.081 Å and c = 15.119 Å [6.3]. Figure 6.7 a is a two-beam bright field image of a particle of 6H-SiC at the foil edge taken using the diffracting vector g=TT20. Thickness fringes visible along the foil edge are due to the wedge shape of the foil. The two-beam diffraction condition with the deviation parameter (s) positive is shown in Figure 6.7 b. EDS spectra of the particle (not shown) indicated that the particle was rich in Si and C.
Figure 6.6: (a), (b) and (c) [001], [013] and [114] zone axes of Si respectively, (d) Kikuchi map for Si near the [001] zone axes showing the spatial relation of the three zone axes in (a), (b) and (c).
Figure 6.7: (a) Two-beam bright field image recorded near the [1\overline{1}00] zone axis of 6H-SiC using $g = \overline{1}120$. (b) Diffraction condition for the bright field image shown in (a). $s > 0$. 
Again, tilting studies were performed to verify the presence of 6H-SiC. Figure 6.8 a is an SAD pattern of the [2110] for the 4H polytype of SiC. While Figures 6.8 b, c and d are SAD patterns that were consistently indexed as the [2110], [1010] and [9091] zone axes of 6H-SiC. Note that the characteristic increased intensity of the 0004 and 0006 reflections in the [2110] SAD patterns of the 4H and 6H polytypes respectively allows easy distinction of the two polytypes. Measured angles between the three axes corresponded very closely with published and calculated angles providing further corroboration. Figure 6.8 e is a section of the Kikuchi map for 6H-SiC near the [2110] zone axis that shows the spatial relation of the three zone axes in reciprocal space.

The [2110], [1010] and [9091] SAD patterns of 6H-SiC are repeated in Figures 6.9 a, b and c to allow comparison with the same patterns calculated using the Desktop Microscopist program and shown in Figures 6.9 d, e and f. The exact matching of the experimentally determined patterns and the calculated patterns provides further proof of the presence and identity of the 6H-SiC in the base material.

Several SiC particles were found to exhibit stepped interfaces with the α-Al matrix. Figure 6.10 a is a multi-beam bright field image recorded near the [1010] zone axis (Figure 6.10 b) of one such particle. The presence of fringes along the vertical faces indicate that these faces are not parallel to the incident electron beam. Conversely, the absence of fringes along the horizontal faces suggest that they faces parallel to the electron beam. Tilting of the sample by 30° along the 0006 Kikuchi band resulted in the position shown by the multi-beam bright field image of Figure 6.10 c taken near the [2110] zone axis. Note the absence of fringes on both faces. These observations suggest that both faces are parallel to the beam in this orientation and that the steps lie on the basal (horizontal) and prism (vertical) planes of the SiC. These steps may be the result of dissolution of SiC along high energy interfaces.
Figure 6.8: (a) [2110] zone axis for 4H-SiC, (b) [2110] zone axis for 6H-SiC, (c) [1010] zone axis for 6H-SiC, (d) [9091] zone axis for 6H-SiC, (e) Kikuchi map for 6H-SiC near the [2110] zone axis showing the spatial relationship between the zone axes shown in (b), (c) and (d).
Figure 6.9: Experimentally determined SAD patterns and calculated SAD patterns (respectively) for: (a) and (b) [2\bar{1}0] zone axis for 6H-SiC, (c) and (d) [10\bar{1}0] zone axis for 6H-SiC, (e) and (f) [90\bar{9}1] zone axis for 6H-SiC.
Figure 6.10: (a): Multi-beam bright field image of stepped 6H-SiC recorded near the [10\bar{1}0] zone axis shown in (b). (c) Multi-beam bright field image of stepped 6H-SiC recorded near the [2\bar{1}10] zone axis shown in (d). Fringes seen on vertical faces in (a) are not present in (c) suggesting that the vertical faces correspond to prism planes and horizontal faces correspond to basal planes.
during reaction with the liquid α-Al matrix during casting followed by reprecipitation at lower energy sights as suggested by Handwerker et. al [2.60]. Alternately, they may stem from particle fracture during SiC production.

TEM examination also revealed faulting of some of the SiC particles under certain diffraction conditions, as shown in Figure 6.11. Figures 6.11 a and c are two-beam bright field images of SiC particles that exhibits faulting. Figure 6.11 a was recorded using the diffracting vector g = 0-110 with s>0 as shown in Figure 6.11 b, while Figure 6.11 c was recorded using the diffracting vector g = 1013 with s>0 as shown in Figure 6.11 d. Faulting of another SiC particle is shown at two different magnifications in Figures 6.11 e and f.

Faulting in SiC has been observed by several researchers and results from the passage of widely separated partial dislocations (with b = 1/3 <1010>) on allowed basal planes that introduce regions of the 3C polytype into the 6H SiC [6.4,6.5]. This result is analogous to (but somewhat more complicated than) the formation of FCC stacking faults in HCP crystals. Passage of a partial dislocation stretches and distorts the Si-C bonds near the basal plane. This local disturbance is thought to weaken these bonds due to the sensitivity of covalent bonds to changes in atomic arrangement and bonding direction [6.5]. TEM studies of mechanically tested Al-MMCs with SiC have shown that cracking of the SiC occurs along the stacking faults and that particle fracture is detrimental to the toughness of the MMC [6.5].

TEM is also very useful in identifying minor phases in small samples. In addition to the other phases already discussed, needles of Al3FeSi were found in the base material. This phase is monoclinic (JCPDS file #20-31) with a = 6.12 Å, b = 6.12 Å, c = 41.5 Å and β = 91° [6.6], and identical to that found by Phragmen [6.7]. This phase has also been referred to as
Figure 6.11: (a) and (c) Two-beam bright field images of faulted SiC recorded near the [2TTT] zone axis using \( g = 0T0 \) and \( g = 10T3 \) diffracting vectors, respectively; (b) and (d) Diffracting conditions for (a) and (c), respectively; (e) and (f) Multi-beam bright field images of SiC.
$\text{Al}_9\text{Fe}_2\text{Si}_2$ [6.8]. Figure 6.12 a is a multi-beam bright field image of a needle of $\text{Al}_3\text{FeSi}$ near the foil edge, while Figure 6.12 b is an EDS spectrum taken from the needle. Quantification of several EDS spectra give an average Al content of $\sim 70 \text{ at. } \%$, average Si content of $\sim 15 \text{ at. } \%$ and an average Fe content of $\sim 15 \text{ at. } \%$. These percentages closely match the 5:1:1 stoichiometric ratio of $\text{Al}_3\text{FeSi}$.

The presence of $\text{Al}_3\text{FeSi}$ was unequivocally determined by completing tilting experiments to identify three different zone axes. Figures 6.13 a-c are SAD patterns that were consistently indexed with the [100], [210] and [60T] zone axes of $\text{Al}_3\text{FeSi}$. Figure 6.13 d is a section of the Kikuchi map for $\text{Al}_3\text{FeSi}$ that shows the relationship between the three zone axes in reciprocal space.

6.2 Comparison of the Laser Beam Welds and Electron Beam Welds

All of the weld schedules produced sound welds with no evidence of porosity or cracking. As expected, the weld cross-section areas increased with increasing power with each of the weld processes. In general, the distribution of SiC in the welds was much more uniform than in the base material due to the rapid cooling rates of the welds. On average, the weld cross-section areas for the LB welds were approximately 25% smaller than that for EB welds made at identical powers, travel speeds and focus conditions. Furthermore, the LB welds were somewhat wider than the comparable EB welds. The differences in weld cross-section areas and aspect ratios for the LB and EB welds can be rationalized by laser plasma effects. During high power LBW, vaporization and ionization of substrate material result in the formation of a plasma in the beam path above the weld pool [6.9]. The plasma acts to attenuate the laser beam by scattering, absorption and defocusing mechanisms and reduce the efficiency of energy transfer to the substrate. Similar attenuation processes do not operate during EB welding because of the lower pressures associated with vacuum EBW. Con-
Figure 6.12: (a) Multi-beam bright field image of Al$_2$FeSi phase: (b) EDS spectrum from needle shown in (a).
Figure 6.13: (a), (b) and (c): the [100], [210] and [60\bar{1}] zone axes of Al\textsubscript{x}FeSi; (d) Kikuchi map for Al\textsubscript{x}FeSi near the [100] zone axis showing the spatial relation of the three zone axes shown in (a), (b) and (c).
sequently, less power reached the substrate for the LB welds relative to the EB welds, and smaller volumes of substrate were melted.

Figures 6.14 a and b are representative optical micrographs of transverse sections of LBW-1 and EBW-1. Recall that these welds were made at identical powers, travel speeds and focus conditions. LBW-1 was slightly wider and shallower than EBW-1. Additionally, observe that the fusion zones of LBW-1 exhibited at least two microstructurally distinct regions, while EBW-1 did not. Specifically, the LBW contains a spike-shaped region along the weld centerline in which little SiC can be observed. Regions immediately adjacent to this spike-shaped region also appeared to be depleted in SiC. These feature were not seen in the EBW. The microstructural differences are discussed in the following sections.

6.3 CO$_2$ Laser Beam Welds

6.3.1 Optical Microscopy and Scanning Electron Microscopy

Figures 6.15 a-c are optical micrographs from LBW-1. The fusion zones of the CO$_2$ LB welds exhibited three distinct regions. Figure 6.15 a and b. The region along the centerline of the CO$_2$ LB welds (Region 1) was shaped like a spike and encompassed less than 25 $\%$ of the weld cross-section. Figure 6.15 a. Region 1 contained large amounts of a black, needle shaped phase in the Al alloy matrix but little SiC. right side of Figure 6.15 b. The Al alloy matrix exhibited a fine cellular structure (~3 $\mu$m spacing). The cooling rate through the solidification temperature range was estimated at approximately 6000$^\circ$ C/s by extrapolating the data of Lloyd and Chamberlain to a 3 $\mu$m primary dendrite arm spacing [2.8]. Examination of the Al alloy matrix with OM and SEM/EDS suggests that it experienced hypoeutectic solidification. (This needle-like black phase is identified as Al$_4$C$_3$ in the TEM/AEM results section of this paper.) Si-rich blocks, identified by EDS data from the SEM.
Figure 6.14: (a) Optical micrograph of EBW-1: (b) Optical micrograph of LBW-1.
Figure 6.15: (a) Low magnification optical micrograph of LBW-1. The dotted line indicates the location of the fusion boundary. Note the microstructurally different regions: (b) Optical micrograph showing the three microstructurally distinct regions: (c) Optical micrograph of Region 1 shown in (b).
appear to be attached to the Al₄C₃ needles. (The Si-rich blocks are identified as Si in the TEM/AEM results section.) The average Si content of the Al alloy matrix of Region 1 of the LB welds was determined to range from 9 to 14 (±0.5%) by weight by SEM/EDS.

The Al₄C₃ in Region 1 was not continuous with any SiC and was relatively long (> 50 µm). Furthermore, the needles were randomly oriented and had some curvature. The needles of Al₄C₃ in Region 1 became scarcer and finer with increasing distance from the top surface of the sample, and more SiC remained. The volume fraction of Al₄C₃ near the top surface of Region 1 was measured at 20.5% with a standard deviation of 5.0 % by examination of several fields at 200x and 400x magnification with the optical image analyzer.

Region 1 of the CO₂ LB welds was surrounded by a very narrow region (Region 2) in which the SiC had partially reacted to form Al₄C₃ and Si according to the displacement reaction. center of Figure 6.15 b. This partially reacted zone (PRZ) did not contain blocks of free Si, and solidification of the matrix in this region appears to have occurred as a hypoeutectic alloy with a Si content close to the nominal matrix composition. The growth of tiny needles of Al₄C₃ from the SiC in the PRZ are evident in Figure 6.15 b. These needles were straight, relatively short, and appear to show an orientation relation with the SiC. (An orientation relation has been determined and is discussed the TEM results section.)

Surrounding the PRZ of the CO₂ LB welds was a large region (Region 3) in which the SiC appeared unreacted (left side of Figure 6.15 b). The unreacted zone (URZ) comprised approximately 70 % of the total weld cross-section. The volume fraction of SiC in Region 3 was determined at 17.4 % with a standard deviation of 2.6 % using the optical image analyzer. Based on the measured volume fraction of SiC and examination using OM and SEM, the matrix of the URZ solidified as a hypoeutectic alloy with a Si content very close
to that of the matrix. Finally, note the SiC depleted areas of Region 3 immediately adjacent to the boundary with Regions 1 and 2. The local volume fraction of SiC appears smaller than that in the remainder of Region 3.

6.3.2 X-ray Diffraction

Figure 6.16 is a plot of an x-ray diffraction spectrum from a sample comprised of several 3 mm diameter disks containing LB welds as well as material from the HAZ and base material. The plot gives intensity as a function of the diffraction angle, $2\theta$, and the spectrum covers diffraction angles from $10^\circ$ to greater than $70^\circ$. The peaks were found to correspond to $\alpha$-Al, Si, 6H-SiC and $Al_4C_3$. Three very small peaks ($2\theta = -28.7^\circ, 29.6^\circ$ and $34.5^\circ$) could not be indexed. The peak at $2\theta = -28.7^\circ$ may correspond to the (020) of $Al_4FeSi$. However, since this was the only peak found for this phase and the intensity of the peak was small, it is not entirely clear if the phase is present.

6.3.3 Transmission Electron Microscopy

The BF micrographs of Figure 6.17 have been obtained from the Al alloy matrix of a thin foil taken near the centerline of the LB welded sheets. The micrograph shows details of the intercellular region between the Al cells which ranged from 2 to 5 $\mu$m in size. Figures 6.17 a. This figure depicts the intercellular region between three $\alpha$ aluminum cells (labels Al$_1$-Al$_3$). The SAD pattern shown in Figure 6.17 b was obtained from the strongly diffracting cell (Al$_1$) in Figure 6.17. Figure 6.17 c is an SAD pattern for an [100] zone axis for FCC aluminum. The streaking in the [010] and [001] along the directions suggest the presence of $\beta''$ precipitates [2.28].
Figure 6.16: X-ray diffraction spectrum for laser beam welds on A356/SiC/15p. Spectrum acquired using Cu K$_\alpha$ radiation. Major peaks were found to correspond to Al, Si, 6H-SiC and Al$_4$C$_3$. 
Figure 6.17: (a) Bright field image of the Al alloy matrix of a LB weld showing three
α-Al grains with intercellular Si. The image was recorded using $g = 111$ for the grain
labeled Al1. (b) Diffraction condition for (a), (c) [001] zone axis for α-Al. Streaking
suggests the presence of β'' precipitates. (d) EDS spectrum for α-Al.
Figure 6.17 d is a representative EDS spectra for the α aluminum. The standardless EDS analyses determined the presence of small amounts of Mg and Si (between 1.1-1.4 at.% Mg and 1.7-1.9 at.% Si) in the Al cells. While these compositions cannot be interpreted in a truly quantitative manner, because the EDS data presented here has not been standardized, it is clear that the α-Al cells of the alloy matrix in the LB welds contain minor amounts of Si and Mg solute. Note that the error in the quantification of the compositional EDS analyses has been determined to be ±0.5 at.%. SAD and EDS analyses of the intercellular regions identified it to contain primarily Si saturated with Al solute. Figure 6.17 a.

The results of the TEM analysis of the black needle-like phase previously observed in the optical micrographs of Figure 6.15 are summarized in Figure 6.18. The two-beam BF micrograph of Figure 6.18 a has been obtained (by exciting two-beam conditions the diffracting vector \( g=0006 \) near the \([10\bar{1}0]\) for \( \text{Al}_4\text{C}_3 \), see below) from a foil centered in Region 1 of Figure 6.15 b. This figure shows a needle of the phase embedded in the surrounding matrix which is comprised of Al cells with intercellular Si and intermetallic compounds. The needle-shaped features were found to be internally faulted with plate-like faults perpendicular to the direction of the \( g \)-vector indicated in Figure 6.18 a. Ledges were observed along the interface with the matrix which is inclined with respect to the incident electron beam in Figure 6.18 a.

The multiple beam BF micrograph of Figure 6.18 b has been obtained for a different zone axis orientation (near the \([2\bar{1}0]\) for \( \text{Al}_4\text{C}_3 \), see below). For this orientation, the interface between the needle and the matrix is edge-on and parallel to the electron beam. The ledges discussed above are clearly seen in this figure. The presence of these features may connote that the growth of \( \text{Al}_4\text{C}_3 \) occurs by advancement of ledges.
Figure 6.18: (a) Two-beam bright field image of a needle of Al₄C₃, embedded in the Al alloy matrix of a LB weld. The image was recorded near the [2110] zone axis of Al₄C₃ using g = 0006. The basal planes are parallel to the beam direction in this image. Growth ledges on the basal planes are indicated by the arrows.; (b) Multi-beam bright field image of another needle of Al₄C₃, showing the growth ledges.; (c) and (d) Experimentally determined SAD pattern and calculated SAD pattern for the [2110] zone axis of Al₄C₃.; (e) and (f) Experimentally determined SAD pattern and calculated SAD pattern for the [1010] zone axis of Al₄C₃.
EDS data representative of the needle-shape features indicated the presence of a majority of Al with significant amounts of C and much smaller amounts of Si. Figures 6.18 c and e are SAD patterns obtained from a needle from Region 1. The tilt angle required to traverse between the two zone axes was approximately 30°. These SAD patterns were indexed consistently with the [2\overline{1}00] and [10\overline{1}0] zone axes for a rhombohedral structure with lattice parameters a=3.33 ± 0.03 Å and c=25.03 ± 0.03 Å (in a hexagonal basis). These observations suggest that the phase is Al₄C₃ (space group R3m, JCPDS file #35-799) which has been shown to have lattice parameters a=3.339 Å and c=24.996 Å in a hexagonal basis [6.10] and may contain small amounts of Si. Figures 6.18 d and f are calculated SAD patterns of the [2\overline{1}00] and [10\overline{1}0] zone axes of Al₄C₃. The exact matching of the experimental and calculated SAD patterns confirms the presence of Al₄C₃.

Further tilting experiments were performed to corroborate the identity of the needle-shaped features. Figure 6.19 a is a section of a Kikuchi map for Al₄C₃, while Figures 6.19 b, c and d are SAD patterns that are consistent with the [\overline{5}50\overline{1}], [\overline{3}4\overline{1}1] and [1\overline{3}2\overline{1}] zone axes of Al₄C₃, respectively. The three SAD patterns were obtained by tilting along the Kikuchi line corresponding to the (0\overline{1}1\overline{5}), as shown in the Kikuchi map. The consistent identification of these zone axes further confirms the presence of Al₄C₃. Note that no evidence of 2Al₄C₃·SiC (Al₈SiC₇), Al₄C₃·SiC (Al₄SiC₄) or Al₄C₃·2SiC (Al₄Si₂C₅) was found in the LB welds described in this work.

As mentioned earlier, the Al₄C₃ was found to exhibit some type of faulting. Figures 6.20 a and b are two-beam bright field images of faulted Al₄C₃. The faults tend to run parallel to the long axis of the needle, and the TEM data suggests that the faults lie on the basal. These faults may result from stresses that are generated during cooling from the weld cycle due to differences in CTE between the Al alloy matrix and the Al₄C₃ and may effect the mechanical properties of the Al₄C₃ in a fashion similar to that discussed for SiC.
Figure 6.19(a) Kikuchi map near the [2110] zone axis of Al₄C₁ from a LB weld showing the spatial relationship of the zone axes given in (b), (c) and (d); (b), (c) and (d) the [5501], [3411] and [321] zone axes of Al₄C₁, respectively.
Figure 6.20: (a) and (b) two-beam bright field images of faulted Al$_4$C$_3$ from a LB weld. Diffraction conditions for the images are shown in the insets.
In Figure 6.21 a, a two-beam BF micrograph centered on an Al₄C₃ needle is shown which has been obtained with diffracting vector \( g = [1\bar{1}1] \) in Si and an incident beam direction approximately \( 6^\circ \) from \([\bar{1}21] \). CBED patterns similar to that presented in Figure 6.21 b were obtained for the various Si grains adjacent to the Al₄C₃. The tilts required to center the beam on the \(<112>\) type zone axes for the various Si grains along the length of the Al₄C₃ needle were always smaller than \( 3^\circ \), which implies that these grains have very similar orientations with respect to the Al₄C₃ needle. The very limited range of orientations detected for the diamond cubic Si grains in contact with the rhombohedral Al₄C₃ needles is consistent with a crystallographic orientation relationship between the two.

Further evidence for an orientation relationship between the Si and the Al₄C₃ needles is given in the two-beam BF micrograph and centered aperture DF micrograph pair shown in Figures 6.21 c and d. The pair of micrographs were obtained using the diffracting vector \( g = [1\bar{1}1] \) for the wedge-shaped piece of Si on the left side of the micrographs. The similar contrast of the second piece of Si on the right side of each micrograph again implies a similar orientation with the Al₄C₃ needle.

A third set of tilting experiments was conducted to establish the exact orientation relationship. Figures 6.22 a and b are indexed microdiffraction patterns for the \([2\bar{1}10]\) zone axis of the Al₄C₃ needle and the \([011]\) zone axis of the adjacent Si, respectively. Figures 6.22 c is an SAD pattern for the same position and tilt with the aperture straddling the interface. Reflections from both the Si and the Al₄C₃ needle can be clearly seen. The orientation relationship determined from this information is: \([\bar{2}110]\) Al₄C₃ \(/\!/ [011] \) Si and \((000\bar{3})\) Al₄C₃ \(/\!/ (\bar{1}\bar{1}1) \) Si. The SAD pattern calculated using the OR above, shown in Figure 6.22 d, matches very closely and confirms the orientation relation.
Figure 6.21 (a) Two-beam bright field of Al$_4$C$_3$ with attached Si from a LB weld recorded with $g = 1\bar{1}1$ for Si.; (b) Convergent beam electron diffraction pattern for Si.; (c) and (d) Bright field/centered aperture dark field image pair of Al$_4$C$_3$ with attached Si. BF image recorded with $g = 1\bar{1}1$ for Si. Dark field image recorded using $g = 1\bar{1}1$ reflection for Si. Note both pieces of Si are "bright" in DF image suggesting similar orientations with respect to the Al$_4$C$_3$. 

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Figure 6.22: (a) Selected area diffraction patterns of [2110] zone axis for Al₄Cₓ from a LB weld: (b) Selected area diffraction patterns of [011] zone axis for attached Si: (c) Selected area diffraction patterns of interface between Al₄Cₓ and Si showing overlap of SAD patterns shown in (a) and (b): (d) Calculated SAD pattern of orientation relation between Al₄Cₓ and Si.
As shown previously in Figure 6.15 b, the growth of tiny needles of Al₄C₃ from the SiC was apparent in region 2 of the LB welds (the PRZ). These needles were straight, relatively short, and appear to show an orientation relation with the SiC. Figure 6.23 a is a two-beam bright field image from Region 2 of an LB weld that shows small needles of Al₄C₃ that appear to have grown from a particle of SiC. The image was recorded near the [1100] zone axis of the largest needle using the diffraction vector $g = 11\overline{2}12$. The diffraction condition is shown in Figure 6.23 b. The orientation relation between the SiC and the Al₄C₃ is determined below in section on EB welding. Figure 6.23 c is a multi-beam bright field image of a SiC particle that appears to have experienced a more extensive reaction. The entire interface appears to have undergone reaction, and needles several microns in length extend into the matrix.

In addition, two intermetallic phases were found in Region 1 of the LB welds: Al₈Si₆Mg₃Fe and Al₃FeSi. The multi-beam bright field image shown in Figure 6.24 a gives an overview of an Al₄C₃ needle surrounded by grains of $\alpha$-Al and Si as well as the two intermetallics. Figure 6.24 b is a multi-beam bright field image of the Al₃FeSi surrounding the right end of the an Al₄C₃ needle in Figure 6.24 a, while Figure 6.24 c is an SAD pattern that has been consistently indexed with the [10\overline{2}] zone axis of Al₃FeSi (monoclinic, JCPDS file #20-32) with $a = 17.8\,\text{Å}$, $b = 10.25\,\text{Å}$ and $c = 8.90\,\text{Å}$ with $\beta = 132^\circ$ [6.11]. Modeling of the SAD patterns for Al₃FeSi was not possible since the atomic positions required to “build” the crystal in the Desktop Microscopist program have not been published.

Figure 6.24 c is a representative EDS spectrum taken from the Al₃FeSi phase. Quantification of several spectra yielded an average Al content of $\sim 73$ at. %, average Si content of $\sim 11$ at. % and an average Fe content of $\sim 14$ at. % with minor amounts of Mg. These percentages do not match closely the 3:1:1 stoichiometric ratio of Al₃FeSi. For example, the
Figure 6.23 (a) Two-beam bright field image of Al₄C₃ needles growing from interface with SiC from a LB weld recorded near the [1100] zone axis using $g = 1 \bar{1} 2 12$ diffracting vector for Al₄C₃; (b) Diffraction condition for (a); (c) Multi-beam bright field image showing larger needles of Al₄C₃ growing from SiC.
Figure 6.24: (a) Multi-beam bright field image of Al₄C₃ needle with attached Si and intermetallics and surrounding α-Al grains from a LB weld. (b) Multi-beam bright field image showing the end of an Al₄C₃ needle with attached Al₅Fe Si intermetallic. (c) Selected area diffraction pattern of the [102] zone axis of the Al₅Fe Si intermetallic. (d) EDS spectrum of Al₅Fe Si intermetallic.
Al content is well above that for the stoichiometric compound. The increased Al content is thought to stem from excitation of x-rays from nearby Al grains.

The $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ also appeared to be attached to the $\text{Al}_4\text{C}_3$ needle. Figure 6.25 a is a multi-beam bright field image of the $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ phase adjacent to the $\text{Al}_4\text{C}_3$ needle. Figure 6.25 b is an EDS spectrum from the $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ phase. Quantification of several spectrum yielded an average Al content of $\sim$56 at. %, an average Si content of $\sim$23 at. %, an average Mg content of $\sim$13 at. %, and % and average Fe content of $\sim$8 at. %. These values deviate somewhat from the exact stoichiometric ratio of the $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ phase.

The presence of $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ was confirmed by completing tilting experiments to identify three zone axes. Figures 6.26 a-c are SAD patterns that have been consistently indexed with the $[2\bar{1}\bar{1}0]$, $[\bar{4}2\bar{2}3]$ and $[2\bar{1}\bar{3}]$ zone axes of the hexagonal $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ phase (P6$_2$m, JCPDS file #25-1085) with $a = 6.62$ Å and $c = 7.92$ Å [6.12]. Angles measured between the three zone axes corresponded closely with calculated and published values map for $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$. Figure 6.26 d is a section of the Kikuchi map for $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ centered near the $[2\bar{1}\bar{1}0]$ zone axis that demonstrates the relationship between the three zone axes in reciprocal space. The SAD patterns of the $[2\bar{1}\bar{1}0]$, $[\bar{4}2\bar{2}3]$ and $[2\bar{1}\bar{3}]$ zone axes are repeated in Figures 6.27 a-c. Figures 6.27 d-f are SAD patterns of the same three zone axes for $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ calculated using the Desktop Microscopist program. The close matching confirms the identification of the $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Fe}$ phase.
Figure 6.25: (a) Multi-beam bright field image showing an Al$_4$C$_3$ needle with attached Al$_2$Si$_6$Mg$_3$Fe intermetallic from a LB weld.; (b) EDS spectrum of the Al$_4$Si$_6$Mg$_3$Fe intermetallic.
Figure 6.26: Experimentally determined and calculated SAD patterns for the (a) and (b) [2\bar{1}0\bar{1}], (c) and (d) [4\bar{2}33] and (e) and (f) [2\bar{1}13] zone axes of Al\textsubscript{x}Si\textsubscript{y}Mg\textsubscript{z}Fe\textsubscript{w}, respectively.
Figure 6.27: (a), (b) and (c) [2110], [4223] and [2113] zone axes of Al₈Si₅Mg₂Fe from a LB weld, respectively; (d) Kikuchi map near the [2110] zone axis of Al₈Si₅Mg₂Fe.
6.4 Electron Beam Welds

6.4.1 Optical Microscopy and Scanning Electron Microscopy

All of the EB welds contained some small amount of Al₄C₃, which was mainly confined to the top center of the fusion zone. Both the number and size of the individual needles of the Al₄C₃ decreased with distance from the top center of the EB welds, but the EB welds did not exhibit distinct regions like the CO₂ LB welds. No Al₄C₃ was evident in the outer region of the EB welds.

Figure 6.28 a is an optical micrograph of the top center portion of the fusion zone of EBW-1. Very little Al₄C₃ was evident anywhere in the fusion zone of the EB welds even at higher optical magnifications (Figure 6.28 b), although one or two small clusters of Al₄C₃ were observed in many of these welds. The microstructure of the EB welds contained a distribution of unreacted SiC particles in an Al alloy matrix with an apparent cell spacing of ~3 μm, as shown in Figure 6.28 c and d. Again, the cooling rate through the solidification temperature range was estimated at approximately 6000°C/s [2.8]. SEM/EDS was used to determine that the Al alloy matrix contained approximately 7 wt. % Si. A volume fraction of SiC of 17.4% with a standard deviation of 3.1% was determined from analysis of images from the top center of EBW-1 weld.

The total volume of Al₄C₃ for EBW-1 and EBW-2 welds was very small. The Al₄C₃ present was very fine (less than 5 μm in length) and was distributed into small (a few hundreds of μm in size) pockets devoid of SiC. The pockets may have resulted from “hot-spots” in the electron beam during welding [6.13]. The SEM was also used to examine the interface between the A356 matrix and the SiC particles in the outer region of the fusion zone of the EB welds where no Al₄C₃ was observed using optical microscopy. No needles
Figure 6.28: (A) Low magnification optical micrograph of EBW-1; (b) Higher magnification optical micrograph of EBW-1. Note the uniform distribution of the SiC and the lack of Al\textsubscript{4}C\textsubscript{3}. (c) Still higher magnification optical micrograph of EBW-1 showing fine solidification cell spacing.; (d) TEM multi-beam bright field image of EBW-1 showing $\alpha$-Al cells with intercellular Si.; (e) Optical micrograph of EBW-3 (defocused weld) showing a large volume fraction of Al\textsubscript{4}C\textsubscript{3}; (f) Optical micrograph of EBW-4 (50 ipm weld) showing a large volume fraction of Al\textsubscript{4}C\textsubscript{3}.
of $\text{Al}_4\text{C}_3$ were visible as either a reaction layer at the interface of the SiC particles or in the matrix during observation of this region with the SEM at magnifications up to 20,000X.

Microstructures of EBW-3 and EBW-4 were somewhat different than those of EBW-1 and EBW-2. Recall that EBW-3 was produced with conditions similar to EBW-2 with the exception of a slightly defocused beam, while EBW-4 was produced with comparable conditions but at a slower travel speed (50 ipm) than EBW-2. Figure 6.28 e is an optical micrograph recorded near the top center of EBW-3. Observe that while some SiC is retained, the microstructure is dominated by the presence of a large volume fraction of $\text{Al}_4\text{C}_3$ needles. Similar results were found with EBW-4. Figure 6.28 f is an optical micrograph from the top center region of EBW-4. Again, note that the microstructure contains a large volume fraction of $\text{Al}_4\text{C}_3$ needles.

6.4.2 Transmission Electron Microscopy

Some of the particles of the SiC in the EB welds were found to contain dislocations. Figure 6.29 a is a two-beam bright field image of dislocations in SiC in an EB weld recorded near the $\langle 2201 \rangle$ ZA of the SiC using the diffracting vector $g=\overline{11}20$. The diffracting condition is shown in Figure 6.29 b. Rows of straight, parallel dislocations appear to emanate from the matrix/SiC boundary. A second set of longer dislocations appear normal to the first set. Determination of the nature and Burgers vector for these dislocations was outside of the scope of this study. They are, however, similar in location and appearance to $1/3<1\overline{1}00>$ partial dislocations observed in SiC by several other researchers [6.5, 6.14].

Faulting was also observed in the SiC in the EB welds. Figure 6.30 a is a two-beam bright field image of faults in SiC in one of the EB welds. The image was recorded near the $[10\overline{1}0]$ ZA with $g=00018$. The diffracting condition is shown in Figure 6.30 b. As dis-
Figure 6.29: (a) Two-beam bright field image of dislocation in SiC near the Al/SiC interface in an EB weld recorded near the [2201] zone axis using $g = \overline{1}120$. (b) Diffraction condition for (a) with $s > 0$. 

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Figure 6.30: (a) Two-beam bright field image of faulted SiC in an EB weld recorded near the [10\bar{1}0] zone axis using $g = 00018$. (b) Diffraction condition for (a) with $s>0$. 

500 nm
cussed earlier, the presence of faults has been related to SiC particle fracture and decreased fracture toughness of Al-MMCs with SiC [6.5].

The orientation relation between the SiC and Al₄C₃ in regions of partially reacted SiC was determined using a sample from an EB weld. Figures 6.31 a and b are many beam bright field images of partially reacted SiC with needles of Al₄C₃ from an EB weld. In both images, the Al₄C₃ appears to form and grow along certain crystallographic directions relative to the SiC.

EDS and tilting experiments were performed to determine the orientation relation between the SiC and Al₄C₃. Figure 6.31 c is an EDS spectrum showing the needle-shaped phase to be rich in C and Al. Figure 6.32 a is a multi beam bright field image of Al₄C₃ needles forming from the SiC. A series of SAD patterns was generated by tilting to a major zone axis for the SiC and recording patterns first with the aperture over the SiC then with the aperture over the Al₄C₃. Figure 6.32 b is an SAD pattern from the [2TT0] zone axis for 6H-SiC. A tilt of less than 2° was required to realign the beam directly with the [2TT0] zone axis of Al₄C₃. Figure 6.32 c. Similarly, the [10T0] zone axis of 6H-SiC. Figure 6.32 d. was closely aligned with the [10T0] zone axis of Al₄C₃. Figure 6.32 e. Hence, the results shown in Figure 6.32 b and c suggest that the orientation relation is very close to: [2TT0] 6H-SiC // [2TT0] Al₄C₃ and (0001) 6H-SiC // (0001) Al₄C₃. Alternately, the orientation relation from Figure 6.31 d and e is very close to: [10T0] 6H-SiC // [10T0] Al₄C₃ and (0001) 6H-SiC // (0001) Al₄C₃. Note that neither OR is exact and that the first OR is crystallographically equivalent to the second due to symmetry.

Figure 6.33 a depicts the calculated superposition of the [2TT0] zone axis of 6H-SiC and the [2TT0] of Al₄C₃ and represents the first orientation discussed above. Similarly, Figure
Figure 6.31: (a) and (b) Multi-beam bright field image of $\text{Al}_4\text{C}_3$ needles emanating from SiC particles from a EB weld.; EDS spectrum from the $\text{Al}_4\text{C}_3$ needles.
Figure 6.32: (a) Multi-beam bright field image of Al$_4$C$_3$ needles emanating from a SiC particle from an EB weld.; (b) and (c) the [2100] zone axes of SiC and Al$_4$C$_3$, respectively from either side of the interface.; (d) and (e) the [1010] zone axes of SiC and Al$_4$C$_3$, respectively, from either side of the interface.
Figure 6.33: (a) Superimposed SAD patterns from the [2\bar{1}0] zone axes of SiC and Al$_4$C$_3$ (calculated with Desktop Microscopist program) showing first orientation relation. (b) Superimposed SAD patterns from the [10\bar{1}0] zone axes of SiC and Al$_4$C$_3$ (calculated with Desktop Microscopist program) showing second orientation relation.
6.33 b shows the superposition of the [1010] zone axis of 6H-SiC and the [10\overline{1}0] of Al₄C₃ in accord with the second orientation relation mentioned above. The second OR is consistent with those reported by other researchers who also found an inexact OR [6.15, 6.16]. Using high resolution electron microscopy (HREM), Yano et. al also showed the presence of a thin transition layer as well as misfit dislocations along the interface [6.16].

6.5 Summary and Conclusions

Results of the microstructural characterization of the base material as well as the LB and EB welds on the cast A356/SiC/15p MMC have been discussed. Study of the welds involved characterization of the fusion zone only. No characterization of the weld heat-affected-zone (HAZ) was undertaken. The solidification cell size of the welds was found to be much smaller than that of the base material owing to the higher solidification rates of the welds. In general, the distribution of SiC in the welds was also much more uniform than in the base material due to the rapid cooling rates of the welds. On average, the weld cross-section areas for the LB welds were approximately 25% smaller than that for EB welds made at identical powers, travel speeds and focus conditions. Furthermore, the LB welds were somewhat wider than the comparable EB welds. Additionally, the microstructure of the LB welds were found to differ considerably from the EB welds in terms of the presence of SiC. The LB welds were found to contain much more of the undesirable Al₄C₃ phase relative to the EB welds. The fusion zone of the LB welds contained three microstructurally distinct regions, while the fusion zone of the EB welds did not. The microstructural information is summarized below in Table 6.1. Knowledge of the microstructures of the EB and LB welds are used along with rationale developed in the next two chapters to develop an understanding of microstructural evolution in the welds.
<table>
<thead>
<tr>
<th>Phase</th>
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<th>EBW</th>
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<th>LBW-3</th>
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<td><strong>SEM/EDS</strong></td>
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<td></td>
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<td></td>
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<tr>
<td>α-Al (wt%)</td>
<td>92.6</td>
<td>91.5</td>
<td>86.9</td>
<td>92.2</td>
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<tr>
<td>Si (wt%)</td>
<td>7.0 ±0.2</td>
<td>8.5 ±0.6</td>
<td>13.1 ±3.5</td>
<td>7.8 ±0.4</td>
</tr>
<tr>
<td><strong>Image Analysis</strong></td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>α-Al (vol%)</td>
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<td>NA</td>
<td>48.5</td>
<td>NA</td>
</tr>
<tr>
<td>Eutectic (vol%)</td>
<td>NA</td>
<td>NA</td>
<td>21.0</td>
<td>NA</td>
</tr>
<tr>
<td>Si (vol%)</td>
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<td>NA</td>
<td>10.6 ±0.8</td>
<td>NA</td>
</tr>
<tr>
<td>SiC (vol%)</td>
<td>16.8 ±4.1</td>
<td>17.1 ±3.4</td>
<td>&gt;2</td>
<td>18.1 ±2.5</td>
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<tr>
<td>Al₄C₃ (vol%)</td>
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<td>&gt;2</td>
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<td><strong>TEM</strong></td>
<td></td>
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<td>NA</td>
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<td>NA</td>
</tr>
<tr>
<td>Al₅FeSi</td>
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<td>NA</td>
</tr>
<tr>
<td>Al₉Si₆Mg₃Fe</td>
<td>No</td>
<td>NA</td>
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</table>

Table 6.1: Results of microstructural characterization.
CHAPTER 7

ENERGY TRANSFER EFFECTS

In the previous chapter, microstructures of the EB welds, LB welds and base material were characterized and discussed. Recall that EB welding resulted in formation of much less undesirable \( \text{Al}_4\text{C}_3 \) phase relative to LB welding at identical power densities and travel speeds. Microstructural development in the different welds appears to hinge on the stability of the Al alloy/SiC interface during the local thermal cycles. Apparently, some physical attribute of the LB process promoted formation of the \( \text{Al}_4\text{C}_3 \) phase during welding. Both EBW and LBW are high-power density welding processes, however there are at least two important differences between the two processes that may contribute to the differences in microstructure in this application. The thermal cycles experienced by the Al alloy/SiC interface may differ during LBW and EBW due to (1) the different processes of energy transfer to the different phases of the substrate material for the two welding processes and (2) the effect of vapor pressure on weld pool temperatures (EBW is performed in vacuum while LBW is performed near atmospheric pressure). The effects of vapor pressure/temperature relations are addressed in the next chapter. The ramifications of the differences in energy transfer for the two processes are discussed in this chapter along with the thermophysical properties of the Al and SiC phases.
7.1 Beam/Substrate Energy Transfer Mechanisms

An interesting observation was made during initial LB welding trials on the Al-MMC. Due to a limited supply of MMC material, weld schedule development was performed on a monolithic Al alloy. However, these weld schedules gave vastly different results when applied to the Al-MMC. Instead of producing acceptable welds, these welding conditions promoted drilling with considerable spattering on the Al-MMC. This observation suggested that the absorptivity of the Al-MMC for the laser energy was considerably larger than that of the monolithic alloy and gave the first clues to understanding microstructural development in the LB welds. Al alloys are known to be very reflective to the 10.6 μm radiation of the CO₂ laser. Apparently, the presence of the SiC in the Al-MMC caused a large increase in absorptivity. This observation also prompted the consideration of EB welding for comparison to the LB welds. The ramifications of these ideas are discussed in the following sections.

Peak temperatures at the Al/SiC interface during LBW are likely greater than those found during EBW owing to the fundamentally different mechanisms that govern coupling of the photon (laser) and electron beams to the different phases of the substrate. Energy transfer to a substrate during LBW occurs by interactions between the photons of the laser beam with the electrons of the substrate, while transfer during EBW results from kinetic energy exchange during collisions between the energetic electrons of the beam and the nuclei and electrons of the substrate atoms. Material properties of Al alloys and SiC that control beam coupling efficiency for EBW and LBW and their effects on SiC loss are discussed in the following sections.
7.1.1 Laser Beam Welding

Electromagnetic radiation like that from the CO₂ laser (10.6 μm) interacts primarily with electrons of the valence and conduction bands of the substrate [7.1.7.2]. No interaction occurs on the atomic level since atoms are too massive to respond significantly to the high frequencies. The optical properties of the substrate are determined by details of the electronic band structure of the substrate material. In general, strongly bound electrons will respond weakly to the electromagnetic field of the laser beam, whereas free electrons can be accelerated thereby absorbing energy from the field. Figure 7.1 is a schematic diagram showing electron excitation and relaxation processes that operate in laser-irradiated materials [7.1].

In semiconductors such as SiC, electrons from the valence band can be excited to the conduction band if the energy of the photons is greater than the bandgap energy [7.1.7.2] (process (a) in Figure 7.1). Note that pure SiC is a wide band gap semiconductor (2.2 to 3.3 eV. depending on the SiC polytype), and the photon energy for CO₂ laser radiation is only 0.12 eV. Hence, photons from the CO₂ laser are incapable of exciting electrons into the conduction band of SiC. However, absorption of laser energy can occur in materials with a wide band gap by transitions other than those between the valence band and conduction band. Electron levels may be introduced within the band gap if impurities or other electrically active defects are present in the material. Absorption of laser radiation can occur as a result of transitions to or from these levels within the band gap. Finally, electrons already in the conduction band of semiconductors can also absorb and transfer photon by free-carrier absorption (process (b) in Figure 7.1).

In metals such as Al alloys, free electrons in the conduction band are accelerated by the oscillating electric field of the photons thereby absorbing energy from the field [7.1.7.2]
Figure 7.1: Photon absorption processes (adapted from [7.1])
(process (b) in Figure 7.1). The oscillating free electrons can share their energy with other electrons via electron-electron interactions with a time constant on the order of $10^{-14}$ s. Energy transfer to the lattice occurs by electron-phonon interactions with a time constant of approximately $10^{-11}$ to $10^{-12}$ s. The oscillating electrons can also reradiate energy resulting in reflection from the substrate.

The two quantities that describe optical coupling are the reflectivity, $R$, which may vary from 0 to 1 and the absorption coefficient, $\alpha$, with units of reciprocal length. $R$ can be determined from the following expression \[7.2\]

$$R = \frac{(\hat{n} - 1)^2 + k^2}{(\hat{n} + 1)^2 + k^2} \quad \text{(Eqn. 7.1)}$$

where $\hat{n}$ is the complex index of refraction \[7.2\]:

$$\hat{n} = n + ik. \quad \text{(Eqn. 7.2)}$$

$n$ is the real part of the complex index of refraction and is simply the ratio of phase velocities in vacuum and the material of interest, and $k$ is the the extinction coefficient which describes damping of the light wave. For good conductors, the index of refraction is proportional to the conductivity:

$$n = \frac{\sqrt{\sigma/2\pi\mu\nu}}{\nu} \quad \text{(Eqn. 7.3)}$$

where $\sigma$ is the electrical conductivity, $\mu$ is the magnetic permeability and $\nu$ is the frequency of the laser radiation. The absorption coefficient, $\alpha$, can be determined from the following expression \[7.2\]:

$$\alpha = 4\pi k/\lambda. \quad \text{(Eqn. 7.4)}$$

For laser irradiation, if light of power density $I$ is normally incident on a substrate, then
For laser irradiation, if light of power density $I_0$ is normally incident on a substrate, then the power density at depth $x$ is given by [7.1, 7.2]:

$$\Phi(x) = \Phi_0 (1 - R) e^{-\alpha x}$$  \hspace{1cm} (Eqn. 7.5)

where $R$ and $\alpha$ are the coupling parameters defined above. This equation predicts maximum power absorption at the surface with an exponential decay length equal to $\alpha^{-1}$.

The efficiency of optical coupling is determined by the reflectivity, $R$. Reflectivity is a function of both the incident wavelength and substrate temperature [7.1, 7.2]. Figure 7.2 a is a plot of the normal reflectance for Al alloy at room temperature as a function of wavelength [7.4]. Figure 7.2 b is a similar plot that also contains data for $\alpha$ [7.2]. For metals such as Al, $R$ is relatively large ($>0.9$) at most wavelengths including the 10.6 $\mu$m wavelength of the CO$_2$ laser. $R$ typically falls to a low value below a characteristic wavelength equal to about 0.1 $\mu$m for Al. $\alpha$ is very large over the entire range of wavelengths due to the high density of free carriers in Al. Hence, the characteristic absorption depth is shallow, and laser radiation is absorbed totally in a depth range of a few hundred Å. The reflectivity of Al decreases for all wavelengths with increasing temperature. For example, the reflectivity of Al alloys for 1.06 $\mu$m radiation falls from about 0.98 at room temperature to approximately 0.85 for liquid Al for the same wavelength as shown in Figure 7.2 c [7.1, 7.2].

The absorption depths and reflectivities for semiconductors, such as Si and SiC, are appreciably different from those of metals. Absorption depths are typically much larger with Si and SiC. Figure 7.3 a is a plot of measured spectral reflectance for SiC for normal incidence radiation at room temperature [7.5]. $R$ is between 0.4 and 0.5 for 10.6 $\mu$m radiation for SiC. Figure 7.3 b shows data for values of $R$ and $\alpha$ for Si [7.2]. While the thermal radiative properties of Si are different, Figure 7.3 b is shown to illustrate the much smaller
Figure 7.2: (a) Normal spectral reflectance for Al (adapted from [7.4]): (b) Spectral reflectivity and absorption coefficient for Al (adapted from [7.2]): (c) I-R for Al for 1 μm radiation (adapted from [7.2]).
Figure 7.3: (a) Normal spectral reflectance for SiC (adapted from [7.5]): (b) Spectral reflectivity and absorption coefficient for Si (adapted from [7.2]).
values of $\alpha$ for semiconductors. Figures 7.4 a and b give reflectivity data for SiC as a function of temperature for 10.6 $\mu$m radiation [7.6]. Note that reflectivity decreases (i.e. $1-R$ increases) with increasing temperature, and $R$ can be less than $\sim 0.15$ at higher temperatures. Figures 7.5 a,b and c also give reflectivity data for SiC as a function of temperature for 10.6 $\mu$m radiation [7.7]. Reflectivity's decrease from approximately 0.8 at room temperature to between 0.1 and 0.2 above 1000 $^\circ$ C for normal incidence (i.e. 0$^\circ$ incidence angle) [7.7]

Table 7.1 provides pertinent information relating to interactions of CO$_2$ laser radiation with Al alloy and SiC substrates. Note that the photon energy for CO$_2$ laser radiation is too small to excite electrons across the bandgap for SiC. Also observe that $R$ for Al is much larger that $R$ for SiC and that $R$ for SiC decreases considerably as it is heated. Values of $R$ for SiC can be as low as 0.15 at high temperature

<table>
<thead>
<tr>
<th>Material Property</th>
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<th>SiC</th>
</tr>
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<tr>
<td>Material Type</td>
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<td>semiconductor</td>
</tr>
<tr>
<td>Band Gap (eV)</td>
<td>NA</td>
<td>2.2 - 3.3</td>
</tr>
<tr>
<td>$R$ for 10.6 $\mu$m</td>
<td>0.80 - 0.94</td>
<td>0.20 - 0.50</td>
</tr>
<tr>
<td>$\alpha^{-1}$ for 10.6 $\mu$m</td>
<td>0.01 $\mu$m</td>
<td>0.1 - 1 $\mu$m</td>
</tr>
</tbody>
</table>

Table 7.1: Material Properties for Important for Interactions with Photons
Figure 7.4: (a) 1-R for SiC for 10.6 μm radiation as a function of temperature for three power levels (adapted from [7.6]); (b) 1-R for SiC for 10.6 μm radiation as a function of time for two power levels (adapted from [7.6]).
Figure 7.5: (a), (b), (c) Reflectivity for SiC for 10.6 μm radiation as a function of incidence angle for three different temperatures (adapted from 7.7).
7.1.2 Electron Beam Welding

Electrons accelerated to high energies interact with the substrate via "collisions" that occur during various scattering events. Scattering involves interactions or collisions between beam electrons and the substrate atoms and electrons that result in changes in the trajectory and/or energy of the incident electrons. The notion of the probability or cross-section of an event is useful in discussing scattering. The cross-section for a scattering event can be defined as [7.8]:

\[ Q = \frac{N}{n_t n_i} \]  \hspace{1cm} (Eqn. 7.6)

where \( N \) is the number of events per unit volume (\( \text{cm}^{-3} \)), \( n_t \) is the number of target sites per unit volume (\( \text{cm}^{-3} \)) and \( n_i \) is the number of incident electrons per unit area (\( \text{cm}^{-2} \)). Hence, the cross-section has units of events/incident electron/(target particle/cm²) or simply (cm²).

The cross-section can be thought of as the effective size of a target atom for a certain type of interaction. Substrates that are dense or contain large atoms thus provide for increased cross-section for interactions and facilitate greater energy transfer to the substrate.

Electron scattering can be divided into two broad categories: elastic and inelastic. Figure 7.6 is a schematic illustration of electron scattering processes. Elastic scattering involves changes in the direction of the incident electron, but the magnitude of the electron velocity is virtually unchanged so that the kinetic energy is nearly constant. Figure 7.6 a. Elastic scattering results from interactions of the beam electron with the nuclei of the substrate atoms, partially screened by their bound electrons. The cross-section for elastic scattering is described by the Rutherford model [7.8]:

\[ Q(> \phi_o) = 1.62 \times 10^{-20} \left( \frac{Z}{E} \right)^2 \cos^2 \left( \frac{\phi}{2} \right) \]  \hspace{1cm} (Eqn. 7.7)
Figure 7.6: (a) Schematic illustration for elastic collisions: (b) Schematic illustration for inelastic collisions (adapted from [7.8]).
where $Q$ is the probability of a scattering event exceeding a given angle $\phi$, $Z$ is the atomic number of the scattering atom and $E$ is the energy of the incident electrons (keV). Note the strong dependence of cross-section on $Z$ and $E$. The cross-section is proportional to the square of the ratio of atomic number and the beam energy ($Z/E$). Consequently, elastic scattering is more probable in materials with larger $Z$ and at lower beam energies.

Inelastic scattering involves transfer of energy from the incident electrons to the target atoms and electrons. Hence, the kinetic energy of the beam electron decreases. Inelastic scattering results from the interaction of beam electrons with electrons of the substrate atoms [7.1.7.2]. There are a number of inelastic scattering processes. But the two most important processes for EBW include plasmon excitation (waves in the “free-electron gas”) and excitation of phonons (lattice waves or heat). The incident electrons transfer energy to the electrons of the substrate in a series of electron-electron collisions. Subsequent electron-phonon interactions allow transfer of energy to the lattice.

Inelastic scattering occurs by a number of different processes. With a variable amount of energy transfer depending on the strength of event. It is often helpful in performing calculations of energy loss to use a “continuous energy loss” term that accounts for all of the inelastic processes. According to Bethe [7.8], the energy loss for inelastic events per unit distance traveled in the solid, $dE/dX$, is given by the expression:

$$
\frac{dE}{dx} = -7.85 \times 10^4 \left( \frac{Z \rho}{A E_m} \right) \ln \left( \frac{1.66 E_m}{J} \right)
$$  \hspace{1cm} (Eqn. 7.8)

where $e$ is the charge of an electron, $N_A$ is Avogadro's number, $Z$ is the atomic number, $A$ is the atomic weight, $\rho$ is the density, $E_m$ is the energy of the incident electrons and $J$ is the mean ionization potential. The continuous energy loss expression, $dE/dX$, has units of keV/
Note that dE/dX increases linearly with Z and \( \rho \). This expression is useful for determining the amount of energy lost by a given beam electron as it travels through the substrate. The variable \( x \) is the distance along the trajectory of the electron and is normally not a straight line.

Elastic and inelastic scattering processes operate simultaneously. Elastic scattering causes changes in direction for the beam electrons and promotes a spreading of electron trajectories through the substrate. Inelastic scattering progressively lowers the energy of the beam electron until it is finally captured by the substrate. The region over which the beam electrons interact with the substrate and thereby deposit energy is termed the interaction volume.

Geometries of interaction volumes have been determined for low beam powers and a low atomic number substrates [7.8]. The volume can be several microns in size and is distinctively pear-shaped. The interaction volumes for EBW of Al/SiC-MMCs are likely larger in size. The size and shape of interaction volumes can also be estimated with Monte Carlo simulations in which the trajectory of an electron is followed through a series of interactions until the electron energy falls below a certain threshold. The process is repeated between 1,000 and 10,000 times to achieve statistical significance.

Monte Carlo calculations for substrates with different Z show that for a constant beam energy the dimensions of the interaction volume decrease with increasing atomic number. This result stems from the strong dependence of \( Q \) on \( Z \) (\( Q \sim Z^{-1} \)) for elastic scattering. With substrates of high atomic number, the beam electrons undergo more elastic scattering per distance and the mean scattering angle is greater. Hence high atomic number substrates tend to promote lower penetrations into the substrate.
Some portion of the beam electrons that strike a substrate will subsequently escape. The reemerging beam electrons are termed backscattered electrons. The backscattered electron coefficient, \( \eta \), is defined as the number of backscattered electrons divided by the number of incident beam electrons.

\[
\eta = \frac{n_{BS}}{n_B} \tag{Eqn. 7.9}
\]

The ratio of the backscattered current to the beam current gives an equivalent value.

Backscattered electrons normally result from a series of elastic scattering events, and hence their energy is not transferred to the substrate. In general, \( \eta \) is found to increase with increasing atomic number as shown in Figure 7.7 a. but is not a strong function of beam energy [7.8].

In analogy to optical reflectivity, an electronic reflectivity, \( R_{el} \), related to the backscattering coefficient may be defined. The power density deposited at a depth, \( x \) is given by the expression [7.1]:

\[
\Phi(x) = \Phi_0 (1 - R_E) f \left( \frac{x}{x_p} \right) \tag{Eqn. 7.10}
\]

where \( R_E \) is the reflection coefficient and \( f \left( \frac{x}{x_p} \right) \) is the gaussian spatial energy deposition profile. As discussed earlier, the energy loss depends on the energy of the beam electrons and the atomic number, \( Z \), of the substrate. Monte Carlo simulations result in an approximately Gaussian distribution of energy loss with the maximum energy loss at depth \( x \). Figure 7.7 b shows a comparison of Monte Carlo results for Al at low beam energy. Note that maximum energy loss occurs at some depth and not at the surface as with LBW. Electrons likely penetrate to a depth of several microns or more into the substrate.
Figure 7.7: (a) Backscattering coefficient for electrons as a function of atomic number; (b) electron energy loss as a function of substrate depth (adapted from [7.8]).
Table 7.2 provides pertinent information relating to interactions of electron beams with Al and SiC substrates. Notice that Z and p are similar for SiC and Al and that R and $\alpha^{-1}$ are also similar. Consequently, no preferential absorption of energy by the SiC occurs during EBW, and the Al and SiC are heated more uniformly.

7.2 Comparison of EBW and LBW

Table 7.3 provides a comparison of factors important to CO2 LBW and EBW. The particles of interest are the photons of the laser beam and the electrons of the electron beam. The energy of the electrons is several orders of magnitude larger than the photon energy. Photons interact with the valence and conduction electrons of the substrate, while the electrons interact with the electrons of the substrate via inelastic scattering processes and with the nuclei of the substrate atoms during elastic events. Coupling is governed by the electronic structure for LBW, while coupling is a function of cross-section for EBW which in turn is dictated by density and atomic number.
Material Property | CO₂ LBW | EBW
--- | --- | ---
Interacting Particles | valence e⁻s & phonons | all e⁻s & nuclei
Particle Energy | 0.12 eV | 15 keV
Properties For Coupling | electronic structure | density, atomic #
Depos. Energy Function | exponential | Gaussian
$\alpha^{-1}$ for Al | 0.01 μm | several μm
$\alpha^{-1}$ for SiC | 0.5 μm | several μm
R for Al | 0.80 - 0.94 | 0.1
R for SiC | 0.20 - 0.50 | 0.1

Table 7.3: Comparison of Material Properties for CO₂ LBW and EBW interaction

The dissipated energy function differs for LBW and EBW. Figure 7.7 is a schematic of the spatial profiles of deposited energy for EBW and LBW. As discussed earlier, the spatial profile for laser beam interaction shows the highest energy absorption at the substrate surface and an exponential decrease in absorption with distance. However, the spatial profile for electron beam interaction is nearly Gaussian in shape with the greatest energy absorption at the position $x_0$. Note that $R$ for Al is much larger than $R$ for SiC for CO₂ LBW and that both are larger than $R$ for EBW. Finally, also note that $\alpha^{-1}$ is much larger for EBW than for LBW.

7.3 Thermophysical Properties

Table 7.4 summarizes the important thermophysical properties of Al and SiC. Note that these properties vary as a function of temperature. Values given in Table 7.4 are representative values. Specific heat, $C_p$, is the energy required the temperature of given mass one degree (cal/gm °C). The thermal conductivity, $k$, is the heat flow per unit area of thermal
Figure 7.8: Comparison of photon and electron absorption (adapted from [7.1]).

LASERS

\[ I(x,t) = I_0(t)(1-R)e^{-ax} \]

ELECTRONS

\[ I(x,t) = I_0(t)(1-R)I \left[ \frac{(x-x_p)^2}{2\sigma^2} \right] \]

WHERE

\[ f_E \propto \exp \left[ \frac{-\left(\frac{x-x_p}{\sigma}\right)^2}{2} \right] \]
gradient (watt/m °C). The thermal diffusivity, D, is given by \( \frac{k}{\rho C} \) and is a measure how large a temperature rise will occur for a given beam/substrate interaction time (cm²/s). It also indicates how rapidly heat diffuses through the substrate. A substrate with low thermal diffusivity will experience little heat penetration, while a high thermal diffusivity substrate will undergo deeper heat penetration.

<table>
<thead>
<tr>
<th>Material Property</th>
<th>Al (A356)</th>
<th>SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting/Liquidus Temp.</td>
<td>625° C</td>
<td>2700° C</td>
</tr>
<tr>
<td>Density (( \rho ))</td>
<td>2.73 gm/cm³</td>
<td>3.21 gm/cm³</td>
</tr>
<tr>
<td>Heat Capacity (( C_\rho ))</td>
<td>0.2 cal/gm°C</td>
<td>0.1 - 0.2 cal/gm°C</td>
</tr>
<tr>
<td>Thermal Conductivity (( k ))</td>
<td>200 - 400 cal/s•cm°C</td>
<td>0.1 - 0.2 cal/s•cm°C</td>
</tr>
<tr>
<td>Thermal Diffusivity (( D ))</td>
<td>(~630) cm²/s</td>
<td>(~1\times10^{-2}) cm²/s</td>
</tr>
<tr>
<td>Diffusion Distance (( d^* ))</td>
<td>(~0.01) cm</td>
<td>(~2.5) cm</td>
</tr>
</tbody>
</table>

Table 7.4: Thermophysical Properties of Al and SiC

Consider a comparison of the thermal diffusivities of Al and SiC. The thermal diffusivity of Al is several orders of magnitude larger that that of SiC. Hence, the heat rapidly penetrates through the Al alloy matrix. The thermal diffusivities define characteristic diffusion distances for a given interaction time. The characteristic diffusion distance is given by the expression:

\[
d = \sqrt{4D\tau}
\]  

(Eqn. 7.11)

where \( \tau \) is the interaction time. For an interaction time of 5 ms, the characteristic thermal
diffusion distance is approximately 0.01 cm for SiC and 2.5 cm for Al. In other words, the heat diffuses much further in the Al alloy matrix in a given time.

7.4 Summary and Conclusions

In the previous chapter, microstructures of the EB and LB welds were characterized. EB welding resulted in formation of much less undesirable Al₄C₃ phase relative to LB welding at identical power densities and rapid travel speeds. In this chapter, the physics of beam/substrate interactions for laser and electron beams with substrates of Al alloys and SiC has been reviewed with a view toward understanding the profound differences in the microstructures between the EB and LB welds. In addition, the thermophysical properties of Al alloys and SiC that control transport of heat have been discussed.

Microstructural development in the different welds appears to hinge on the stability of the Al alloy/SiC interface during the local thermal cycles. A comparison of the material properties governing beam/substrate interactions (Table 7.3) may hold the answer to the differences in the LBW and EBW microstructures. While exact values for thermal radiative properties of the Al alloy and the SiC that comprise the Al-MMC studied here are not known, approximate values can be determined from the published values for similar materials discussed above. The discrepancy between the reflectivities for Al and SiC for LBW is consistent with preferential energy absorption by SiC particles that encounter the laser beam during welding. Essentially, the Al alloy reflects a large fraction of the incident laser energy, while the SiC locally absorbs a large fraction of the energy. Moreover, the reflectivity of the SiC drops rapidly as it is heated and it becomes an even stronger absorber of CO₂ laser radiation. Energy transfer from the laser beam to the SiC occurs over a time constant several orders of magnitude shorter than the time required for appreciable thermal diffu-
sion. Consequently, the SiC is heated rapidly by the laser beam during the interaction time resulting in high peak temperatures at the Al/SiC interface. In a sense, the energy transfer is dominated by the SiC phase during LBW, and this condition may lead to "hot-spots" around the SiC particles. These high temperatures appear to drive reaction and dissolution of the SiC which result in subsequent formation of Al$_4$C$_3$ upon solidification.

Because the density and atomic number for the Al alloy are similar to the density and average atomic number of SiC, there is no preferential absorption of electron beam energy by either phase and heating is thought to be more spatially uniform during EBW of this MMC. Due to a larger $\alpha^4$, absorption also occurs over a larger interaction volume with EBW relative to LBW. Heating of the SiC occurs over a much longer time constant that with LBW, and heating of the SiC is limited by the high thermal diffusivity of the Al phase in contact with the SiC. Consequently, the energy transfer is dominated by the Al phase.

In the next chapter, the effects of vapor pressure/temperature relations on the microstructural differences between the LB and EB welds are discussed.
CHAPTER 8

VAPOR PRESSURE-TEMPERATURE EFFECTS

The microstructures of the EB and LB welds were characterized and discussed in Chapter 6. The EB welds exhibited greater retention of the SiC reinforcement relative to the LB welds. To the contrary, the central region of the LB welds contained a large volume fraction of the undesirable Al$_4$C$_3$ phase. Two rationalizations have been offered for the differences in microstructures. The first involves effects of beam/substrate energy transfer and was discussed in Chapter 7.

The second rationalization deals with pressure-temperature relations. Peak temperatures during LBW and EBW may also differ due to pressure-temperature effects. LBW is performed near atmospheric pressure, while EBW is normally accomplished in a partial vacuum (~10^-4 torr). The effects on peak temperatures experienced by the Al/SiC interface due to the differences in pressure are discussed in this chapter.

8.1 Keyhole Formation

Both LBW and EBW are capable of producing power densities (>10^9 W/m^2) in excess of those required for vaporization from liquid metal weld pools. This vaporization results in a recoil force which causes depression of the weld pool surface and in turn allows increased
deposition of beam energy to greater depths. The increase in incident beam energy promotes greater vaporization, and the process is self-accelerating. Ultimately, a stable vapor cavity called a “keyhole” may be established, as shown in Figure 8.1. Keyholing allows for very deep and narrow weld profiles. Keyholes will not form below a certain threshold power density for vaporization. The vapor cavity issurrounded by a column of liquid metal (or in this case liquid Al and SiC/AlC). The vapor cavity along with the column of liquid advances with the beam, while the beam interacts with the weld pool at the liquid/vapor interface along the leading surface of the keyhole. The high depth-to-width ratio of many of EB and LB welds produced in this study suggests that they were keyhole welds.

Stability of the keyhole relies on the balance of forces tending to close it and those tending to maintain it [8.1]. Figure 8.2 is an idealization of the pressure balance in a static keyhole. The pressures that act to stabilize the keyhole include the beam pressure ($p_b$), the vapor pressure ($p_v$) and the recoil pressure ($p_r$), while the gravitational pressure ($pgh$) and surface tension pressure ($2\gamma/r$) tend to close the keyhole.

For LBW, the beam pressure is the radiation pressure and is equal to $W/Ac$ (Eqn. 8-1) where $W/A$ is the power density and $c$ is the speed of light. For EBW, the beam pressure is given by the electron pressure and is equal to $(2J^2m/v/e)^{1/2}$ (Eqn. 8-2) where $J$ is the current density and $v$ is the beam voltage. Considerable vaporization occurs along the leading edge and bottom of the keyhole. The vapor pressure thus generated acts against the keyhole walls and also tends to open the keyhole. The gravitational pressure results from maintenance of the hydrostatic (actually metallostatic) head of the column of liquid metal surrounding the vapor cavity. The gravitational pressure is given by $pgh$ and acts to close the keyhole. The surface tension pressure given by $2\gamma/r$ (Eqn. 8-3) for a keyhole with a hemispherical bottom also tends to close the keyhole.
Figure 8.1: Schematic of a keyhole (adapted from [8.1]).

Figure 8.2: Schematic of an idealized stationary keyhole showing pressure balance (adapted from [8.1]).
A pressure balance for a stable keyhole can be considered [8.1]. The beam pressure, recoil pressure and vapor pressure are balanced by the hydrostatic head and surface tension pressure. Hence, the pressure balance may be written as:

$$P_b + P_r + P_v = \frac{2\gamma}{r} + \rho gh$$  \hspace{1cm} (Eqn. 8-4)

or

$$h = \frac{\left(P_b + P_r + P_v - \frac{2\gamma}{r}\right)}{\rho g}$$  \hspace{1cm} (Eqn. 8-5)

where $h$ is the depth of the keyhole. More importantly, note that there is a pressure gradient along the depth of the hole with pressure gradient dropping to zero near the top of the keyhole. The pressure at the bottom of the keyhole is

$$p_b = p_i + \Delta p$$  \hspace{1cm} (Eqn. 8-6)

where $p_i$ is the ambient pressure and $\Delta p = P_b + P_r + P_v = 2\gamma r + \rho gh$. This $\Delta p$ must be present to maintain a keyhole for an EB or LB weld. Note that the ambient pressure for EBW is a partial vacuum. Consequently, the pressure at the bottom of an EB keyhole can be less than one atmosphere. On the contrary, the pressure at the bottom of an LB keyhole is slightly above one atmosphere.

8.2 Vapor Pressure-Temperature Relations for Aluminum Alloys

The equilibrium vapor pressure of a gas in contact with a condensed phase is related to temperature by some form of the Clausius-Clapeyron equation. One form of the relation-
ship for pure aluminum is given by [8.2]:

\[
\log p(\text{torr}) = \frac{-16380}{T} - \log T + 12.32. \tag{Eqn. 8-7}
\]

At equilibrium, this relation places an upper bound on the melt surface temperature of 2450\(^\circ\)C at one atmosphere of pressure and 1240\(^\circ\)C at a pressure of 10\(^{-4}\) torr.

The vapor pressure-temperature relation for Al is modified by alloying. Evaporation of alloying elements with high vapor pressures from Al alloys during welding has been reported by several groups of researchers [8.3-8.6]. Furthermore, the presence of relatively volatile elements, such as Mg and Zn have been found to change the equilibrium vapor pressure and hence the temperature of the weld pool surface [8.3-8.6]. Equilibrium pressure-temperature (P-T) diagrams have been developed for several Al alloys [8.3]. A P-T diagram for Al-Mg alloys and other alloying elements is reproduced in Figure 8.3. The dashed line in Figure 8.3 indicates the pressure-temperature relation for a 0.4 \% Mg alloy (similar to A356). The equilibrium surface temperature of a melt of 0.4 \% Mg alloy predicted by the P-T diagram is greater than 2000 \(^\circ\)C at one atmosphere of pressure and well below 1000\(^\circ\)C at 10\(^{-4}\) torr (~10\(^{-7}\) atm).

Block-Bolten and Eagar calculated peak temperatures for stationary arc welds on several Al alloys using a model that combined thermodynamic data with the kinetic theory of gases [8.3]. They reported a range of maximum possible temperatures from 1615 \(^\circ\)C to 2090 \(^\circ\)C for welds made on nominally pure Al (1100) and from 1360 \(^\circ\)C to 1880 \(^\circ\)C for welds produced on an Al-Mg-Si alloy (6061). Using a similar approach, Cieslak and Fuerschbach determined peak temperature ranges for CW Nd:YAG laser beam (LB) welds on 6061 and several Al-Mg alloys [8.4]. They estimated that the peak temperature of the
Figure 8.3: Vapor pressure-temperature relations for several Al alloys. Dashed line indicates P-T relation for 0.4 % Mg alloy. (adapted from [8.3]).
welds produced on 6061 is between 820° C and 1290 °C. Metzbower employed a somewhat similar approach to determine temperature distributions in the keyhole of LB welds on Al-Mg alloys [8.5]. This model predicts peak temperatures of approximately 2500° C at the center of the keyhole for pure Al alloys. However, predicted peak temperatures drop to the range from 2000° C to 1650° C with increasing Mg content.

Schauer et al. measured peak surface temperatures in the keyhole of electron beam (EB) welds using an infrared pyrometer [8.6]. Representative peak temperatures of 1900 ± 100° C were reported for welds made on 1100 and 1800± 100°C for welds on 6061. The temperature values reported by Schauer et al. are considerably higher than the limiting value of about 1240° C predicted by the vapor pressure-temperature relationship for melts produced in a partial vacuum. Concerns have been raised as to whether Schauer et al. were actually measuring weld pool temperatures or the temperature of the vaporized/ionized substrate material escaping from the keyhole. Hence, these results may be inaccurate.

8.3 Pressure-Temperature Effects During EBW and LBW

The P-T diagrams and models discussed above are based on thermodynamics and are strictly valid only at equilibrium. The thermal cycles of LB and EB welds like those produced in this study are rapid and transient in nature, and the weld pool temperatures may not achieve equilibrium. The rates of approach to equilibrium for the weld pools are governed by kinetic factors which are difficult to assess. Nonetheless, the P-T diagrams and models may be used to estimate trends in peak temperatures in the welds produced on the Al-MMC used in this work.

The A356 matrix of the composite used for this study is an Al-Mg-Si alloy with approximately 0.4% Mg by weight. The pressure-temperature behavior of the A356 matrix is
expected to fall somewhere between those of pure Al (given by Equation 8-7) and the 0.4% Mg alloy (from Figure 8.3). The thermodynamic models discussed above predict peak temperatures greater than 2000°C for an Al alloy with about 0.4 wt % Mg (like the A356 alloy matrix) at a pressure of one atmosphere. Additionally, analysis of the P-T diagram suggests that, for all other factors constant, peak temperature of welds produced on A356 in a partial vacuum may be significantly lower than the peak temperature of welds made near atmospheric pressure. While there is some disparity in the measured and calculated values for peak temperatures for LB and EB welds on Al alloys, the consensus of research suggests that peak temperature of welds on A356 fall between 1950 and 2450°C at one atmosphere of pressure (like the LB welds) but are limited to a range between 800 to 1240°C in partial vacuum (like the EB welds).

These lower peak temperatures of the EB welds would be expected to result in greater retention of SiC and less formation of Al₅C₃. While SiC is not thermodynamically stable when in contact with liquid hypoeutectic Al alloys, the lower temperatures would limit rates of the dissolution and displacement of SiC since the rates of both processes are both likely proportional to the exponential of negative reciprocal absolute temperature (\( \exp \left[ -1/T \right] \)).

8.4 Pressure-Temperature Effects in Electron Beam Welding

In this chapter and the previous chapter, two rationalizations have been offered for the possible differences in peak temperatures and the resultant differences in microstructures in EB and LB welds on A356/SiC/15p. The first involved effects of beam/substrate energy transfer, while the second dealt with pressure-temperature relations. These two mechanisms may work in concert or one may dominate. In an attempt to discern whether one process dominates peak temperatures, experiments to compare EBW in vacuum (conven-
tional EBW) and at atmospheric pressure (NVEBW) were carried out. As discussed in the chapter on experimental procedures, EB welds and NVEB welds were made at similar powers and travel speeds. Unfortunately, the electron beam rapidly diverged due to scattering at atmospheric pressure. Consequently, the microstructures of the NVEB welds looked much like the defocused EB welds made in vacuum and contained a considerable volume fraction of $\text{Al}_4\text{C}_3$. Hence, this experiment was not able to accurately discern the effects of pressure on temperatures and microstructures in EB and LB welds on A356/SiC/15p. Further work is required to resolve this issue. A possible solution involves comparison of laser welds made in vacuum through a quartz glass window using the Nd:YAG laser process with those made in the same enclosure at atmospheric pressure.

8.5 Summary and Conclusions

In this chapter, the effects on peak temperatures experienced by the Al/SiC interface due to the differences in pressure for EBW and LBW on A356/SiC/15p were discussed. The peak temperature achieved by a melt of liquid metal is related to the ambient pressure via some form of the Clausius-Clapeyron equation. Peak temperatures of melts produced in vacuum are limited to the boiling point of the liquid. As a consequence, the peak temperatures of the EB welds produced in this study under partial vacuum are expected to be considerably lower (by up to several hundreds of °C) than LB welds made at atmospheric pressure at identical powers, travel speeds and focus conditions. The lower peak temperatures associated with EBW are also expected to limit rates of the dissolution and displacement of SiC and allow greater retention of a desired microstructure. Experiments aimed at discerning the effects of pressure on temperatures and microstructures in the EB and LB welds were not successful and further work may be required.
CHAPTER 9

MICROSTRUCTURAL DEVELOPMENT

In this chapter, the microstructural information determined in the characterization phase of this work (Chapter 6) is combined with the beam/substrate energy transfer and pressure-temperature concepts discussed previously (Chapters 7 and 8 respectively) to propose qualitative models for microstructural development of the EB and NVEB welds on the A356/SiC/15p MMC as well as the different regions of LB welds. Differences in microstructures are related to variations of the weld thermal cycle. In the first section of the chapter, microstructural evolution is discussed in terms of estimated time-temperature curves for the different regions and processes. Subsequently, the Al-Si-C ternary phase diagram is employed to develop models for microstructural evolution. The models are compared with the microstructural information found in Chapter 6 and with the results of mass balance calculations.

9.1 Time-Temperature Profiles

The stability of SiC in contact with liquid Al alloys was discussed in the literature review of Chapter 2. At relatively low temperatures (up to \(-1200^\circ\) C), SiC was shown to undergo a displacement reaction to form \(\text{Al}_4\text{C}_3\) when in contact with liquid Al alloys[2.58,2.59]. At higher temperatures (>1900\(^\circ\) to 2000\(^\circ\) C), SiC will dissolve into the liquid solution according to the Al-Si-C ternary phase diagram [2.21]. Welding of the A356/
SiC/15p MMC involved application of a transient thermal cycle capable of melting the Al alloy matrix and providing superheat to the melt pools. The microstructural development of the various regions of the different welds is a strong function of the stability of the SiC during the local thermal cycles. Consistent with the phase diagram, weld thermal cycles with higher peak temperatures will tend to result in dissolution of SiC, while those with lower thermal cycles will produce $\text{Al}_4\text{C}_3$ via the displacement reaction. Moreover, thermal cycles that provide longer times at temperature will allow the dissolution and/or displacement reactions to proceed to a greater extent.

9.1.1 Laser Beam Welds

Figure 9.1 is a schematic illustration of the proposed thermal cycles for the three different regions of the LB welds. Based on the results of the characterization work and the concepts discussed in previous chapters, the thermal cycles for the LB welds appeared sufficient capable of causing dissolution (Region 1) and displacement (Region 2) of the SiC. The microstructure of Region 1 of the LB welds is consistent with a weld thermal cycle in which the SiC is dissolved into the liquid solution at high temperatures and $\text{Al}_4\text{C}_3$ is precipitated from this liquid solution on cooling. Figure 9.1. Due to the high absorptivity of SiC for the 10.6 $\mu$m laser radiation, the SiC particles that encounter the laser beam at the liquid/vapor interface in the keyhole are heated very rapidly. With the high power densities used, the temperature of the surface of the SiC at the liquid/vapor interface may rise as high as its melting/sublimation temperature. The temperature of the liquid Al alloy matrix is likely at or above 1900° to 2000° C. Hence, heat transfer ensues between the hot SiC and the cooler Al alloy matrix. Simultaneously, the surfaces of the SiC in contact with the Al alloy dissolve carrying Si and C into solution. The time available for dissolution of SiC in contact with the liquid Al alloy is great enough to allow complete dissolution for the LB
Figure 9.1: Thermal cycles for the three regions of the LB welds.

Figure 9.2: Comparison of thermal cycles for LB and EB welds.
welds. The $\text{Al}_4\text{C}_3$ forms on cooling by precipitation from the liquid solution. Further details of the microstructural evolution are discussed in Sections 9.2 and 9.3.

Region 2 of the LB welds develops adjacent to the keyhole. SiC particles in region 2 do not interact directly with the laser beam. The presence of SiC particles (as well as $\text{Al}_4\text{C}_3$) in Region 2 caused an increase in melt viscosity during welding and hence limits convective heat transfer. Consequently, Region 2 is mainly heated by conduction from Region 1, and peak temperatures in Region 2 were lower than those experienced in Region 1 as depicted in Figure 9.1. However, the local thermal cycle was sufficient to form $\text{Al}_4\text{C}_3$ by direct displacement of the SiC, and small needles of $\text{Al}_4\text{C}_3$ formed and grew from the SiC. Again, more information on the microstructural evolution is given in Section 9.2 and 9.3.

The melt viscosity in Region 3 was also relatively high owing to the presence of SiC particles throughout the thermal cycle. Region 3 was largely heated by conduction from Region 2, and peak temperatures in Region 3 were lower than those in Region 2, Figure 9.1. The thermal cycle was insufficient to produce evidence of $\text{Al}_4\text{C}_3$. The A356 matrix melts and resolidifies before discernible amounts of $\text{Al}_4\text{C}_3$ can form by either dissolution/reprecipitation or displacement.

9.1.2 Comparison of Electron Beam Welds and Laser Beam Welds

Figure 9.2 is a schematic illustration comparing the proposed thermal cycles of the LB and EB welds. The microstructural evidence from the EB welds made at rapid travel speeds is consistent with a weld thermal cycle in which the Al alloy matrix melts and resolidifies rapidly with little loss of SiC, Figure 9.2. Consideration of the pressure-temperature relations and a lack of preferential energy absorption by the SiC suggest that the interface
between the liquid Al alloy and the SiC experiences a lower peak temperature during EB welding. Hence, peak temperatures experienced by the Al/SiC interface (and perhaps the entire weld pool) were higher for the LB welds as illustrated in Figure 9.2. and the time at temperatures was probably longer. Consequently, the time available for dissolution and/or displacement of the SiC was much less for the EB welds relative to the LB welds. and little Al₄C₃ was formed.

9.1.3 Effects of Defocus. Travel Speed and Pressure on the Electron Beam Welds

Figure 9.3 allows discussion of the effects of defocus, travel speed and pressure on the EB welds. The curve labeled 1 is intended to represent the thermal cycle for an EB weld, such as EBW-1 and EBW-2, made at rapid travel speed (for example 200 ipm) with an electron beam focused at the substrate surface. The curve labeled 2 is intended to represent the thermal cycle for an EB weld produced under one of the following conditions: defocused beam, slower travel speed or at atmospheric pressure. Recall that the fusion zones of the defocused EB welds (EBW-3) and the EB welds made at 50 ipm (EBW-4) contained considerable Al₄C₃. Defocusing of the electron beam and decreasing the travel speed will flatten thermal gradients and will increase the length of time experienced above the eutectic temperature. This effect increased the time available for contact between the SiC and the liquid Al alloy. Hence, while the peak temperatures of the thermal cycles experienced by these types of welds may have been equal to those found in the other EB welds, the times available for displacement and/or dissolution of the SiC were probably longer.

The lack of vacuum caused the electron beam of the NVEB welds to broaden rapidly with increasing distance from the exit orifice causing an unintended defocusing of the beam. Hence, arguments similar to those proposed for the defocused EB welds may be applied to
Figure 9.3: Comparison of thermal cycles for conventional EB welds made at rapid travel speeds with a focused beam and other EB welds.
the NVEB weld. Again, the broader heat source resulted in shallower gradients that allowed
more time for displacement and/or dissolution of the SiC.

9.2 The Al-Si-C Phase Diagram and Microstructural Evolution in the LB Welds

The phases present at different times during evolution of the microstructure and their
compositions can be estimated with the aid of the Al-Si-C ternary phase diagram. Figure
9.4 gives the liquidus projection for this system. Note that compositions are given in atomic
percent. The primary crystallization fields for the various phases are shown along with the
compositions of the A356 matrix, SiC and Al$_4$C$_3$. The minor elements in the A356 matrix
are thought to have little effect on the important features of microstructural evolution and
are ignored in this discussion. The A356 is assumed to have a composition of 93 at. % Al
and 7 at. % Si and is shown near the Al-rich corner of the diagram on the Al-Si binary join.
Similarly, SiC is located at 50 at.% Si and 50 at.% C on the Si-C binary join, while Al$_4$C$_3$ is
found at ~57 at.% Al and ~43 at.% C on the Al-C binary join.

Microstructural evolution in Region 1 of the LB welds on the A356/SiC/15p MMC is
considered first. The A356/SiC/15p MMC is a combination of the A356 alloy matrix and
the SiC reinforcement. As the LB welds are produced, at least some of the SiC in the path of
the beam encounters the laser beam at the liquid/vapor interface of the keyhole. As men­
tioned several times previously in this document, the SiC is thought to dissolve into the
liquid Al alloy solution during heating due to preferential absorption of the laser energy and
the higher temperatures allowed at one atmosphere pressure (relative the the EB welds). As
the SiC dissolves, the composition of the weld pool is enriched in both Si and C. By mass
conservation, the average composition of the weld pool at any time during dissolution of
the SiC must fall along the dashed line shown in Figure 9.4 that joins the compositions of
the A356 matrix and the SiC.
Figure 9.5 shows an enlarged view of the Al-rich corner of the liquidus projection and part of the join connecting the compositions of the A356 matrix and the SiC. As the SiC is dissolved, the average composition of the weld pool moves along the line in the direction shown by the arrows in Figure 9.5. Since the microstructural analysis has shown that no SiC remains in Region 1, the SiC is assumed to dissolve completely, and the composition of the weld pool reaches the position indicated by the X in Figure 9.5 upon complete dissolution of all of the SiC. At this point in time, Region 1 of the weld pool contains a liquid solution of Al, Si and C with small amounts of Mg, Fe and other trace elements and is at temperatures above ~1900°C to 2000°C.

Image analysis has shown the A356/SiC/15p MMC to actually contain 17 vol. % SiC by volume. Mass balance calculations performed assuming complete dissolution of 17 vol. % SiC in an Al-7 at.% alloy yield a composition of 70.0 at. % Al, 17.5 at.% Si 12.5 at.% C for the point marked X in Figure 9.5. Note that this composition falls at a point on the boundary between the primary crystallization fields for C (graphite), Al$_4$C$_3$•SiC (AS) and 2Al$_4$C$_3$•SiC (2AS). Consequently, crystallization (solidification) of one or more of these phases is expected under equilibrium conditions. However, none of these phases were found in the final microstructure of this region of the LB welds. This result is not entirely surprising, though, since the thermal cycles associated with the LB welds made in this study were very rapid, and attainment of equilibrium was not necessarily expected.

On cooling, Al$_4$C$_3$ is apparently the first phase to solidify from the weld pool. As they are curved and relatively long, the needles of Al$_4$C$_3$ are thought to precipitate directly from the liquid Al-Si-C alloy on cooling as opposed to forming from displacement of the SiC. The Al$_4$C$_3$ subsequently appears to grow by extension of growth ledges on the basal plane.
Figure 9.4: Liquidus projection for the Al-Si-C ternary system (adapted from [2.22]).

Figure 9.5: Al-rich corner of the liquidus projection for the Al-Si-C ternary system. The path for dissolution of SiC in the A356 matrix is indicated by the arrowed line (adapted from [2.22]).
$\text{Al}_4\text{C}_3$ is the first phase to form despite the fact that the composition falls far from the primary crystallization field for $\text{Al}_4\text{C}_3$. There are a few possible explanations for this result. Use of the liquidus projection to describe microstructural evolution in this instance requires a homogenous liquid solution in equilibrium at a peak temperature above about 2100° C as estimated from the isotherm lines shown in Figure 9.5. Failure to meet any of these requirements (homogeneity, equilibrium and temperature) will limit the accuracy of results predicted by the liquidus projection. It is possible that peak temperature was not high enough to completely dissolve all of the SiC, or that nucleation of $\text{Al}_4\text{C}_3$ is kinetically favored over the equilibrium phases.

One possible reason for preferred nucleation of $\text{Al}_4\text{C}_3$ may involve the presence of some remnant solid in the liquid solution at peak temperatures. As mentioned earlier, the $\text{Al}_3\text{FeSi}$ and $\text{Al}_8\text{Si}_6\text{Mg}_3\text{Si}$ intermetallics appear to nucleate upon the $\text{Al}_4\text{C}_3$ needles. The opposite scenario should be considered however since the relative solidification temperatures of the $\text{Al}_4\text{C}_3$ and intermetallics under the prevailing conditions are not well known, and the composition of the liquid solution at peak temperature does not fall in the primary crystallization field for $\text{Al}_4\text{C}_3$. Nucleation of the $\text{Al}_4\text{C}_3$ upon the intermetallics is possible. However, the microstructures suggest otherwise. For example, the volume fraction of the intermetallics is too small to allow nucleation of the large number of $\text{Al}_4\text{C}_3$ needles found in the LB welds. Moreover, many of the $\text{Al}_4\text{C}_3$ needles had more than one grain of attached intermetallic. Consequently, the $\text{Al}_4\text{C}_3$ needles likely form first followed by the intermetallics.

It is also possible that the time at temperature was not sufficient to completely dissolve all of the SiC, as mentioned above. Recall the SiC depleted regions adjacent to Region 1 noted in the optical micrographs in Chapter 6. Some of the partially reacted SiC with at-
tached needles of may have been swept into Region 1 behind the laser beam thus avoiding direct contact with the beam. Dissolution of the rest of the partially reacted SiC particles likely continued at high temperatures leaving small needles of $\text{Al}_4\text{C}_3$ in the liquid to act as sites for continued growth of $\text{Al}_4\text{C}_3$ on subsequent cooling. If this scenario occurs, the dissolution path shown in Figure 9.5 is not followed exactly. Since some of the Al and C are consumed in $\text{Al}_4\text{C}_3$ formation, the final composition upon complete SiC dissolution will be slightly richer in Si and leaner in Al and C.

Regardless of the exact details of dissolution and initial precipitation, the composition of the weld pool becomes progressively leaner in C and Al as the $\text{Al}_4\text{C}_3$ solidifies. The composition must follow along a path toward the Al-Si edge of the diagram that parallels the line joining the composition corresponding to $\text{Al}_4\text{C}_3$ and the final composition upon complete dissolution (perhaps 70.0 at. % Al, 17.5 at. % Si and 12.5 at. % C), as shown in Figure 9.6. The composition follows this path until all of the C is consumed in forming $\text{Al}_4\text{C}_3$ and the composition reaches a position very close to the Al-Si edge of the diagram. Mass balance calculations aimed at determining the amount of $\text{Al}_4\text{C}_3$ formed in this fashion give a volume fraction of 21.3 % which is very close to the fraction of 19.5 % measured using image analysis.

Formation of the $\text{Al}_5\text{FeSi}$ and $\text{Al}_9\text{Si}_6\text{Mg}_3\text{Si}$ intermetallics in the LB welds is now discussed briefly. The results of microstructural analysis suggest that these intermetallics nucleate on the $\text{Al}_4\text{C}_3$ needles from the melt during cooling. The intermetallics containing Al, Fe and Si only are considered first. Recall that $\text{Al}_5\text{FeSi}$ intermetallic was found in the base material, while the $\text{Al}_3\text{FeSi}$ intermetallic was observed in the LB welds. The Al-rich corner of the Al-Fe-Si ternary phase diagram is shown in Figure 9.7 [9.1]. As shown in Figure 9.7, the presence of $\text{Al}_3\text{FeSi}$ in the LB welds is consistent with the higher Si level of the melt in the LB welds relative to that of the A356 base material (~17.5 vs. ~ 7%).
Figure 9.6: Al-rich corner of the liquidus projection for the Al-Si-C ternary system. The path for precipitation of $\text{Al}_2\text{C}_3$ from the liquid solution is indicated by the arrowed line (adapted from [2.22]).

Figure 9.7: Al-rich corner of the liquidus projection for the Al-Fe-Si ternary system (adapted from [9.1]).
Upon completion of the formation of the Al₄C₃ and the intermetallics, the composition of the weld pool in Region 1 is very close to an Al-Si binary alloy. Mass balance calculations yield a composition of 75 at.% Al and 25 at. % Si. The Al-Si binary phase diagram shown in Figure 9.8 can be used to illustrate further microstructural evolution of the LB welds. The path followed by the composition during solidification of Region 1 is depicted by the arrowed line on the right of Figure 9.8. As the temperature of the weld pool in Region 1 reaches the liquidus temperature for this composition, the blocky Si seen previously in micrographs begins to nucleate on the Al₄C₃ needles.

Recall that little or no Al₄C₃ was found in Region 3 of the LB welds and the Si content of matrix in the region remained very close to the ~ 7 % found in the base material. The arrowed line on the left side of Figure 9.8 illustrates the path followed by Region 3 of the LB welds. Simultaneous to the precipitation of the Si in Region 1, epitaxial growth of α-Al begins from the partially melted grains along the fusion boundary.

Upon further cooling, the composition of the weld pool in Region 1 follows the hyper-eutectic liquidus line during solidification of the Si as shown by the arrowed line on the right side of Figure 9.9. Solidification of Si in Region 1 causes rejection of Al locally from the melt. Similarly, the composition of the weld pool in Region 3 during cooling follows the hypoeutectic liquidus line during solidification of the α-Al as shown by the arrowed line on the left side of Figure 9.9. Solidification of α-Al in Region 3 promotes rejection of Si from the melt. Solidification of the α-Al continues through Region 3 to Region 2 and finally throughout Region 1 as the solidifying α-Al surrounds and envelopes the preexisting Al₄C₃ needles with attached Si and intermetallics. Finally the two solid fronts reach the eutectic composition and the final solidification product is the Al-Si eutectic.
Figure 9.8: The binary Al-Si phase diagram. The paths for solidification of Regions 1 and 3 of the LB welds are indicated by the arrowed lines (adapted from [2.16]).

Figure 9.9: The binary Al-Si phase diagram. The paths for solidification of Regions 1 and 3 of the LB welds are indicated by the arrowed lines (adapted from [2.16]).
Calculations using the Scheil equation and the simple inverse lever-law coupled with mass balance calculations yield the following volume fractions for the phases present in Region 1 of the LB welds at the end of solidification: 65% Al alloy (α-Al plus eutectic), 13.7% Si and 21.3% Al₄C₃. These values compare closely to the average values determined by image analysis (listed previously in Table 6-1): 69.5% Al alloy (α-Al plus eutectic), 10.6% Si and 19.5% Al₄C₃. Further details of the mass balance calculations and image analysis are given in Appendix B.

Similar rationale can be used to describe microstructural development in the EB welds made with a defocused beam, at slow travel speeds and at atmospheric pressure. However, in the conventional EB welds made at rapid travel speeds with a focused beam, the Si content and volume fraction SiC in the fusion zone was identical to that of the base material. The lower temperatures and rapid travel speeds promote little or no dissolution/displacement of the SiC, and no Al₄C₃ is formed.

9.3 Summary and Conclusions

The microstructural information determined in the characterization phase of this work has been combined with the beam/substrate energy transfer and pressure-temperature concepts discussed previously to propose qualitative models for microstructural development of the EB and NVEB welds on the A356/SiC/15p MMC as well as the different regions of LB welds. Differences in microstructures are related to variations of the weld thermal cycle. Time-temperature curves for the different regions and processes consistent with the microstructures have been developed. Additionally, the time-temperature curves and the Al-Si-C ternary phase have been utilized to propose qualitative models for microstructural evolution of the different regions and processes that are consistent with the results of mass balance calculations.
Briefly, the SiC that encounters the laser beam at the liquid/vapor interface of the keyhole in Region I of the LB welds is thought to dissolve into solution, and the Al₄C₃ and intermetallics precipitate from the liquid solution. The Al₄C₃ in Region 2 develops as a result of partial displacement of the SiC. The α-Al with Al-Si eutectic growing from the substrate in Region 3 of the welds envelops the partially reacted SiC in Region 2 and the Al₄C₃ and intermetallics in Region 1. The SiC in Region 3 is largely unreacted. The NVEB welds contained three distinct zones similar to those of the LB welds. The SiC in the conventional is not preferentially heated by the electron beam, and the partial vacuum limits peak temperatures in the weld pool. Consequently, the A356 matrix melts and resolidifies with no loss of SiC in the EB welds.
Chapter 10

Ultrasonic and Tensile Testing of Cast Plates

As discussed in the Objectives chapter, one of the goals of this work is to determine the mechanical properties of the EB and LB welds produced in this study and to understand the effects of the different microstructures of the EB and LB welds on the mechanical properties. Recall that little work has been done to measure tensile properties of welded Al-MMCs. While many authors have claimed that the presence of $\text{Al}_4\text{C}_3$ in weld and casting microstructures is detrimental to mechanical properties, few studies have actually been performed to test this theory and the little work performed to date has been inconclusive and confusing.

Only two studies applicable to joining of SiC reinforced Al-MMCs have been published. As discussed in the Literature Review chapter, Iseki et al. joined two types of SiC to Al alloys: reaction bonded and sintered [2.58,2.59]. The reaction bonded SiC contained excess Si along grain boundaries, while the sintered SiC did not. During joining, the Si along the grain boundaries of the reaction bonded SiC allowed penetration and wetting of the grain boundaries by the Al alloy. Conversely, no penetration of the grain boundaries occurred during bonding of the sintered SiC. Bend testing of the samples after joining showed that the reaction bonded SiC samples had lower strengths relative to the reaction bonded SiC samples and failed along cracks that developed at the grain boundaries. The
authors claimed that the presence of $\text{Al}_4\text{C}_3$ at the sintered SiC/Al alloy interface actually gave stronger joints than the reaction bonded samples.

The other pertinent study was conducted by Dahotre et al [3.40]. As outlined previously in the Literature Review, they compared mechanical properties of cast base material from large commercial ingots with those of LB welds on the same material. The LB welding conditions used did not allow an accurate comparison of properties. Specifically, some welds were actually intermittent, while others allow for repeated thermal cycling. The authors reported that the laser welds had properties that were superior to those of the base material despite the presence of large amounts of $\text{Al}_4\text{C}_3$. They did not recognize the effects of the disparity in grain size and other microstructural features on the mechanical properties.

Results of these two studies are actually contrary to conventional thinking, and a proper study of mechanical properties that accounts for this disparity is required. As mentioned in the chapter on Experimental Procedures, plates of 359/SiC/10p and 359/SiC/20p Al-MMCs were chill cast to produce fine-grained castings for subsequent welding and mechanical testing. Prior to machining and mechanical testing, the plates were examined nondestructively first visually and then using C-Scan imaging. Subscale tensile bars were then machined from plate sections deemed to contain the least porosity and tested. The rest of this chapter describes the results of the C-scan and tensile testing.

10.1 Results of Ultrasonic C-Scan Testing

The results of the C-scan testing are shown in Figures 10.1, 10.2 and 10.3. Figure 10.1 shows the percent attenuation of the ultrasonic beam as a function of position on several of
Figure 10.1: Ultrasonic C-scan results: percent attenuation versus position for cast plates. Light (green and yellow) areas indicate areas of lesser attenuation corresponding to networks of porosity.
the as-cast plates that contained no defects during visual testing. Greater attenuation occurs on sound, pore-free areas of the castings since the average density of the Al-MMC is greater than that of the porous areas. The dark (purple) areas indicate very little attenuation. The light (green and yellow) areas indicate lesser amounts of attenuation i.e. greater scattering (relative to the orange colored areas) and correspond to areas containing porosity. The plate labeled 10 appears to contain excessive porosity near the center of the plate, while the plates labeled 2, 4 and 7 appear to contain networks of porosity in several areas. Plates 1, 3 and 9 apparently contain a few areas of porosity. The presence of porosity in the light (green and yellow) areas of several plates was verified using optical microscopy of polished metallographic samples.

Figure 10.2 shows the time-of-flight results for the same plates. The lighter gray (yellow) plates correspond to the 359/SiC/10p plates, while the darker gray (orange) plates correspond to the 359/SiC/20p plates. This observation is consistent with the faster speed of sound through the 359/SiC/20p plates due to the higher volume fraction of SiC. Darker (green) areas can be seen on plates 1, 3 and 9 consistent with longer times-of-flight through the porous areas.

Figure 10.3 shows percent attenuation of the ultrasonic beam as a function of position on several machined subscale tensile bars. Note that the tensile bars were thinner than the original cast plates and that the color scale on Figures 10.1 and 10.3 should not be compared directly. Figure 10.3 shows many light (green) colored regions throughout the gage length of the tensile bars corresponding to greater amounts of local scattering and regions of inhomogeneity. The presence of the inhomogeneities caused concern for the quality of the castings and the tensile samples.
Figure 10.2: Ultrasonic C-scan results: time-of-flight versus position for cast plates. Lighter gray (yellow) plates correspond to 359/SiC/10p, while darker gray (orange) plates correspond to 359/SiC/20p. Black (green) areas correspond to porous areas.
Figure 10.3: Ultrasonic C-scan results: percent attenuation versus position for subscale tensile bars. Light (green and yellow) areas indicate areas of lesser attenuation corresponding to networks of porosity.

10.2 Results of Tensile Testing

To determine whether welding experiments on the cast plates would yield useful data, four subscale tensile bars were machined from the cast plates. Two samples were taken from a 359/SiC/10p plate and two were taken from a 359/SiC/20p plate. Tensile testing of the subscale tensile bars gave yield and tensile strengths below those reported by the manufacturer of the Al-MMC. Yield and tensile strengths of the tensile bars averaged ~15 and ~20 ksi respectively for the bars versus ~32 and ~24 ksi reported for castings in the solution annealed condition. The quality of castings of these Al-MMCs are described as sensitive to the casting conditions.
The poor tensile properties of the tensile bars made further testing and welding experiments useless. Failure of tensile bars occurs in the weakest area of the gage length. Tensile tests on welded samples containing porous areas in the gage length would give no useful data on weld strengths. Hence, further welding experiments were abandoned.

10.3 Summary and Conclusions

The cast plates apparently contained a density of defects that rendered the plates useless for further welding trials. Consequently, no measurements of mechanical properties of the welds was possible. Die casting of plates should be considered if higher quality plates are to be produced for welding trials.
CHAPTER 11

SUMMARY and CONCLUSIONS

Laser beam (LB) and electron beam (EB) welds have been produced at sharp focus using identical powers, rapid travel speeds and focus conditions on plates of an A356/SiC/15p MMC. EB welds were also produced using slower travel speeds and defocused beams to assess the effects of these variables. In addition, non-vacuum EB welds were also made. The microstructures of the base material and the fusion zones of the welds have been characterized using optical microscopy (OM), scanning electron microscopy (SEM), x-ray diffraction (XRD) and transmission electron microscopy (TEM) with the goal of developing an understanding of the evolution of the weld microstructures during joining. The following conclusions can be drawn based upon the results of this work.

1) The microstructure of the as-cast base material is characterized by the presence of coarse (~75 μm in size) solidification cells of primary α-Al with intercellular Al-Si eutectic, approximately 17 vol. % 6H-SiC and minor amounts of Al₅FeSi intermetallic. The average Si content of the Al alloy matrix was measured to be ~ 7 at. %, and evidence of faulting was observed in some of the SiC particles.

2) The transverse cross-sections of the fusion zones of the LB welds were 20 to 25 % smaller than those of the EB welds made at identical weld powers, travel speeds and focus conditions. These differences can be attributed to attenuation of the laser beam by the plasma.
that forms during LBW and lowers the power for input to the substrate. Similar attenuation mechanisms do not operate during EBW, and a larger fraction of the beam power reaches the substrate.

3) The fusion zones of the LB welds exhibited three microstructurally distinct regions. Region 1 was situated along the weld centerline, and was shaped like a spike. The area fraction of Region 1 was approximately 20% of the cross-sectional area of the fusion zone. Region 1 was found to contain fine cells (~3 μm) of primary α-Al with intercellular Al-Si eutectic, blocks of primary Si (~10.6 vol. %), needles of Al₄C₃ (~19.5 vol. %) up to ~100 μm in length and minor amounts of Al₃FeSi and Al₈Si₆Mg₃Fe intermetallics. The average Si content of Region 1 ranged from 9 to 14 at. %. The Si blocks and the intermetallics were attached to the Al₄C₃ needles. The following approximate orientation relation was determined for the Al₄C₃ and attached Si blocks: [2110]Al₄C₃ // [110]SiC and (0003)Al₄C₃ // (111)Si.

4) Region 1 of the LB welds was surrounded by Region 2 which contained of primary α-Al with intercellular Al-Si eutectic and partially reacted SiC: i.e. small needles of Al₄C₃ growing from the SiC. No blocks of primary Si were found in Region 2. The area fraction of Region 2 was approximately 5 % of the cross-sectional area of the fusion zone.

5) Region 3 encompassed Regions 1 and 2 and constituted about 75 % of the cross-sectional area of the fusion zone of the LB welds on average. Region 3 contained fine cells (~3 μm) of primary α-Al with intercellular Al-Si eutectic and ~18 vol. % SiC. The average Si content was found to be ~7.8 at. %. Note that the Si and SiC contents in Region 3 are nearly identical to those of the base material.

6) EB welds made at sharp focus position did not contain microstructurally distinct regions. The fusion zones of the EB welds contained fine cells (~3 μm) of primary α-Al with
intercellular Al-Si eutectic and ~ 17.8 vol. % SiC. The average Si content of the EB welds was ~ 7.8 at. % Considerably less Al₄C₃ was found in these welds relative to the LB welds made at identical parameters. The small amounts of Al₄C₃ that were observed were found either as “pockets” of individual needles or as small needles growing from SiC particles. The following approximate orientation relation was determined for the Al₄C₃ growing from the SiC particles: [10\overline{1}0]SiC // [10\overline{1}0]Al₄C₃ and (0001)SiC // (0001)Al₄C₃

7) EB welds produced with one of the following conditions: defocused beam, slower travel speed or in non-vacuum conditions contained distinct microstructural regions similar to those found in the LB welds. However, there was a smaller volume fraction of Al₄C₃ in the region analogous to Region 1 of the LB welds and a larger volume fraction of surviving SiC.

8) Variations in microstructures of the EB and LB welds made using identical powers, travel speeds and focusing conditions were attributed to the differences in the thermal cycles experienced with the two welding processes. In turn, the disparities in thermal cycles were rationalized in terms of: a) the differences in the physics of beam/substrate energy transfer for the two processes and b) pressure temperature relations.

9) The efficiency of optical (laser) coupling for a given wavelength is governed by the reflectivity (R) of the substrate. Reflectivity is a function of the conductivity, temperature and surface condition of the substrate. For Al alloys, R was relatively large (~0.9) for the laser wavelength used in this study, while R for SiC was somewhat lower (~ 0.4 to 0.5). These differences in R for Al alloys and SiC were consistent with strong absorption of the laser energy by the SiC that resulted in rapid heating and dissolution of the SiC.

10) Energy transfer to the substrate during EB welding occurs by kinetic energy exchange between the accelerated electrons of the beam and the atoms and electrons of the substrate
during scattering events. For a given accelerating voltage, the probability for scattering and hence energy transfer is a function of the atomic number and density of the substrate. Specifically, the probability of scattering increases for substrates with higher densities and greater atomic numbers. The important result of this information for this study is that the atomic number and density of SiC and Al alloys are similar, and no preferential energy transfer occurred to the SiC phase. Most of the beam energy was absorbed by the Al alloy matrix since it was the majority phase, and the heat was quickly drawn away from the SiC due to the high thermal diffusivity of the Al. Hence, the SiC was not heated as much as in the LB weld, and less displacement/dissolution of the SiC occurred.

11) A component of the differences in the microstructures between the LB and EB welds may also have occurred due to pressure-temperature relations. LBW was performed near atmospheric pressure, while EBW was completed in a partial vacuum. Peak temperatures during EBW in vacuum may have been several hundreds of degrees Celsius lower than peak temperatures during LBW near atmospheric pressure. Lower peak temperatures were consistent with slower rates of displacement and dissolution of SiC as well as greater stability of the SiC.

12) Differences in the microstructures of the standard EB welds and those made using a defocused beam, at slower travel speeds or without a partial vacuum were attributed to longer thermal cycles that allowed greater time for displacement and/or dissolution of the SiC.

13) Microstructural evolution in Region 1 of the LB welds was consistent with complete dissolution of the SiC into the liquid Al alloy at temperatures of ~ 2000° C followed by precipitation of Al₄C₃, Si and the intermetallics upon cooling. Alpha Al grew epitaxially
from the fusion boundary, through Regions 3 and 2, and enveloped the Al$_4$C$_3$, Si and the intermetallics. Mass balance calculations using this model matched closely the volume fractions and compositions of the phases present in the final microstructures.

14) Microstructural evolution in Region 2 of the LB welds was consistent with moderate amounts of displacement of the SiC by Al$_4$C$_3$ and a small increase in Si content. In Region 3 of the LB welds, the Al alloy melted and resolidified with little or no loss of SiC. The SiC and Si contents of Region 3 were nearly identical to those of the base material.

15) Microstructural evolution in the standard EB welds was similar to that in Region 3 of the LB welds. Conversely, microstructural evolution in the EB welds made using a defocused beam, at slower travel speeds or without a partial vacuum was similar to that in Region 1 of the LB welds.

16) Attempts to measure mechanical properties of the welds were unsuccessful due to the poor quality of the cast plates produced for welding trials.
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Chapter 2


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Chapter 3


Chapter 5


5.3 Desktop Microscopist Program. Virtual Laboratories. Ukiah, CA.

Chapter 6


Chapter 7


Chapter 8


Chapter 9


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APPENDIX A

WELDING PROCESSES

The purpose of this appendix is to provide a brief review of the welding processes used for joining DRA-MMCs. Several processes can be used. There is no single ideal welding process. Each process has inherent advantages and disadvantages in terms of its capability and cost. Welding processes can be conveniently divided into two major categories. Fusion welding processes involve melting of the parent material and filler metal (if used), while no melting occurs during solid-state welding processes.

One distinguishing feature of all fusion welding processes is the intensity of the heat source used for melting. Power densities must be great enough to cause melting of some area as opposed to just heating up the entire workpiece. In simplest terms, the rate of energy flow into a given volume must be greater than the rate at which energy flows out of the volume if melting is to occur. As a rough guide, power densities in excess of 1000 W/cm² are required to melt most metals. The spectrum of power densities for several common welding processes are shown in Figure A1 [A1]. Power densities range from $10^3$ W/cm² for oxy-fuel gas welding to $10^6$ W/cm² for laser and electron beam welding. Power densities above $10^6$ W/cm² tend to promote vaporization of metals in a short period and are not practical for welding.

The high power densities available with laser and electron beam welding allow for the production of deep welds in a single pass. Under these conditions, vaporization of the mate-
rial of the substrate results in the formation of a vapor cavity called a “keyhole” surrounded by a column of liquid. At steady-state, the metallostatic head of the column of liquid is balanced by the force from the vapor pressure at the bottom of the keyhole. The keyhole acts as a blackbody aiding absorption of the beam. The weld is produced by carrying the keyhole along the joint. Fluid flow occurs around the keyhole from the front of the column to the back.

Figure A.1: The Spectrum of power densities for fusion welding processes (reprinted from Reference A1).

A second advantage of high power densities relates to the ability to achieve rapid thermal cycles for non-equilibrium processing. Power density is inversely related to the interaction time required for melting, as shown in Figure A2 [A1]. Materials with a high thermal diffusivity lie near the top of the band in Figure A2, while those with low thermal diffusivity lie near the bottom. A high power density process allows production of a given size of weld at faster travel rates relative to a lower power density process. The fast travel rates promote rapid thermal cycles which may be exploited for limiting undesired reactions during welding of materials such as Al/SiC MMCs. Several welding processes are reviewed in the following...
Friction Welding

Friction Welding (FRW) is a solid-state welding process in which heat is generated by friction from the relative motion of the parts to be welded, as shown in Figure A3. The heat of welding originates from direct conversion of mechanical energy of the moving part(s) to thermal energy and strain energy. The application of an axial force maintains intimate contact of the parts and causes plastic deformation of the material near the interface during
welding. More than 90% of the energy consumed in plastic deformation is transformed into heat, although a small fraction of the energy is stored in the material as strain energy. Deformation is largely restricted to the volume of material adjacent to the original interfaces by an adiabatic shear process. During adiabatic shearing, the temperature rise in this volume results in a local loss in flow stress thereby decreasing the resistance of the material to further deformation. As a result of the lowered resistance, deformation tends to be localized in this volume, and the process continues in a self-propogating fashion.

![Friction Welding Process](image)

Figure A.3: Schematic of the Friction Welding Process (reprinted from Reference A2).

Brazing

Brazing joins materials by heating them in the presence of a filler metal having a liquidus temperature above 450° C but below the solidus temperature of the base material. Hence no melting of the base material occurs. Heat can be provided by a variety of processes. The
filler metal is distributed between the closely fitted surfaces of the joint by capillary action, as shown in Figure A4. Solidification of the filler metal occurs during cooling of the assembly.

![Figure A.4: Schematic of the Brazing Process](reprinted from Reference A1).

**Transient Liquid-Phase Bonding**

Transient Liquid-Phase (TLP) bonding is very similar to brazing. TLP bonding joins metals by heating them to a suitable brazing temperature at which a preplaced filler metal melts and flows by capillary attraction. Unlike brazing, solidification occurs isothermally as a result of diffusion of elements from the filler metal into the base material. Figure A5. Pressure may or may not be applied to aid in joining. The diffusion process often results in the total loss of identity of the original brazed joint.

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Gas Tungsten Arc Welding

Gas Tungsten Arc Welding (GTAW), also known as TIG welding, creates a coalescence of a base material by maintenance of an electric arc between the workpiece and a nonconsumable tungsten electrode. Figure A6. A localized inert atmosphere is provided by a shielding gas which protects both the electrode and the molten weld pool from contamination. GTAW is a very versatile process capable of producing very clean, high quality welds. It is easily automated and capable of welding in all positions on almost any base metal. The process may be utilized with or without additions of filler wire. Different arc polarities may be employed to allow manipulation of the operating characteristics. The process may be operated in the direct current electrode positive (DCEP), direct current electrode negative (DCEN), or alternating current (AC) modes. Alternating current is frequently employed with aluminum base materials for its cathodic cleaning of the oxide layer around the weld area which serves to improve the wetting of the molten weld pool.

Figure A.6: Schematic of the Gas Tungsten Arc Welding Process (reprinted from Reference A2).
Gas Metal Arc Welding

In the Gas Metal Arc Welding (GMAW) process (also known as the MIG welding process), an arc is maintained between a continuous, consumable wire electrode and the workpiece. Figure A7. The electrode serves as filler metal as it is fed through the welding torch from a wire spool. The filler metal is incorporated into the weld pool after it is transferred across the arc. A shielding gas is supplied through the torch nozzle with the purpose of protecting the molten weld pool from the atmosphere. This process is easily automated and offers high deposition rates due to the continuous wire electrode feed. GMAW can be used on most metals and alloys and may employ different arc polarities. For welding of aluminum alloys, alternating current is frequently utilized resulting in the same cathodic cleaning action as in GTAW.

Figure A.7: Schematic of the Gas Metal Arc Welding Process (reprinted from Reference A2).
Capacitor Discharge Welding

Capacitor Discharge Welding (CDW) is an arc welding process in which the tip of a rod or stud is melted by rapid discharge of energy stored in a bank of capacitors. The rod is initially held away from the work surface by the welding head. Figure A8. At the beginning of the weld cycle, the rod is forced toward the work surface using spring pressure or air pressure. Before the rod contacts the work surface, the voltage difference between the two causes initiation of an arc which melts the the tip of the stud and the adjoining work surface. The weld is completed by plunging the rod into the workpiece. The weld cycle time for this process is very short (from 4 to 6 ms), and the penetration of the weld zone into the work surface is normally from 0.10 to 0.15 mm.

![Figure A.8:Schematic of the Capacitor Discharge Welding Process](reprinted from Reference A1).

Flash Welding

Flash Welding (FW) is a resistance welding process in which a butt joint weld is produced by a melting and displacement of surface asperities followed by the application of pressure. The two parts to be joined are held in clamps connected to the secondary of a resistance welding transformer. Figure A9. Voltage is then applied and the parts are slowly moved together. Resistance heating occurs at the points of asperity contact causing rapid melting and expulsion of the material followed by arcing. This process continues until the
faying surfaces are molten and a short length of each part is heated to forging temperature. The weld is completed by termination of the voltage and the application of an upset force to push the surfaces into intimate contact and forge the parts together.

Laser Beam Welding

Laser Beam Welding (LBW) is a fusion welding process that uses a moving high power density (>10^5 W/cm^2) coherent optical energy source called a laser as the source of heat. Figure A10. The coherent nature of the laser beam allows it to be focused to a small spot, leading to high power densities. Two types of lasers are commonly used for welding: the continuous-wave (CW) carbon dioxide (CO_2) laser and the neodymium-doped yttrium aluminum garnet (Nd:YAG) laser. The ability to tailor power and focus the beam allows precise control over the placement and size of the weld. Very small welds with narrow HAZs can be produced. The high power densities available with LBW also allow for rapid thermal cycles for non-equilibrium processing.

Electron Beam Welding

Electron Beam Welding (EBW) is a high power density fusion welding process that is accomplished by bombarding the joint to be welded with an intense (strongly focused) beam of high energy electrons. Figure A11. The instantaneous conversion of the kinetic energy of these electrons into thermal energy as they impact and penetrate into the workpiece on which they are impinging causes melting and produces the weld. Electromagnetic focusing of the electron beams allows power densities in excess of 10^6 W/cm^2 to be obtained. Like LBW, the prime advantages of EBW is the ability to make welds that are deeper and narrower than arc welds, with a total heat input that is much lower than that required in arc welding. This ability to achieve a high weld depth-to-width ratio eliminates the need for multiple-pass welds, as required in arc welding. The lower heat input results in a narrow workpiece heat-affected zone (HAZ) and fewer thermal effects on the workpiece.
Figure A.9: Schematic of the Flash Welding Process (reprinted from Reference A1).
Figure A.10: (a) Schematic of a Solid-State Laser Cavity (b) Schematic of the Laser Beam Welding Process (reprinted from Reference A2).
Figure A.11: Schematic of the Electron Beam Welding Process (reprinted from Reference A2).
References


APPENDIX B

MASS BALANCE CALCULATIONS

The purpose of this appendix is to outline the mass balance calculations for C and Si that are used to corroborate the qualitative models for microstructural evolution. The results of the microstructural characterization showed that the A356 matrix contained approximately 7 wt. % Si and 17 vol % SiC. These values are used as a starting point for the mass balance calculations.

Consider a basis of one cm$^3$ of the A356/SiC/17p MMC. Seventeen percent of this volume is comprised of SiC. SiC has a density of 3.217 gm/cm$^3$. The number of grams of SiC in this volume is:

\[
0.17 \text{ cm}^3 \text{ SiC} \times 3.217 \text{ gm/cm}^3 = 0.547 \text{ gm SiC}.
\]

The molecular weight of SiC is 40.10 gm/mole. Hence, the number of moles of SiC is:

\[
0.547 \text{ gm SiC} \times 1 \text{ mole SiC/40.10 gms} = 1.364 \times 10^{-2} \text{ moles SiC}.
\]

If this amount of SiC dissolves in the liquid Al alloy solution it contributes $1.364 \times 10^{-2}$ moles of Si and $1.364 \times 10^{-2}$ moles of C.
The remainder of the volume of the Al-MMC (83%) is made up of the A356 alloy. The density of the A356 alloy is 2.685 gm/cm$^3$. The number of grams of alloy is:

$$0.83 \text{ gms A356} \times 2.685 \text{ gm/cm}^3 = 2.228 \text{ gms A356}.$$  

The alloy is 93% Al and 7% Si. Thus there are:

$$0.93 \times 2.228 \text{ gms} = 2.072 \text{ gms Al}$$

and

$$0.07 \times 2.228 \text{ gms} = 0.156 \text{ gms Si}.$$

The molecular weight of Si is 28.09 gm/mole and the molecular weight of Al is 26.98 gm/mole. This gives:

$$0.156 \text{ gm Si} \times 1 \text{ mole Si/28.09 gm} = 5.554 \times 10^{-2} \text{ moles of Si}$$

and

$$2.072 \text{ gm Al} \times 1 \text{ mole Al/26.98 gm} = 7.680 \times 10^{-2} \text{ moles of Al}.$$

At high temperatures, the Si and C are dissolved into a homogenous Al alloy that contains: $7.680 \times 10^{-2}$ moles of Al, $1.364 \times 10^{-2}$ moles of C and $1.919 \times 10^{-2}$ moles Si ($1.364 \times 10^{-2}$ moles from the A356 alloy $+ 5.554 \times 10^{-1}$ moles from dissolution of the SiC). This combination yields the following atomic percentages: 70.05% Al, 12.45% C and 17.50% Si at peak temperatures.

On cooling, the C is consumed in forming $\text{Al}_4\text{C}_3$. Specifically, 4 moles of Al combine with 3 moles of C to form 1 mole of $\text{Al}_4\text{C}_3$. Hence:
\[ 1.364 \times 10^{-2} \text{ moles C} \times 4/3 = 1.819 \times 10^{-2} \text{ moles of Al} \]

are consumed. This leaves:

\[ (7.680 - 1.819) \times 10^{-2} \text{ moles of Al alloy} \]

in solution. Additionally:

\[ 1.364 \times 10^{-2} \text{ moles C} \times 1/3 = 4.547 \times 10^{-3} \text{ moles of Al}_4C_1 \]

are formed.

This leaves: \(5.681 \times 10^{-2}\) moles of Al and \(1.919 \times 10^{-2}\) moles Si in solution with \(4.547 \times 10^{-3}\) moles of \(\text{Al}_4C_1\) as precipitates. The total number of moles of alloys is:

\[ 5.681 \times 10^{-2} \text{ moles of Al} + 1.919 \times 10^{-2} \text{ moles Si} = 7.780 \times 10^{-2} \text{ moles of alloy} \]

The composition of the remaining alloy (in atomic percent) is:

\[ \frac{5.681 \times 10^{-2} \text{ moles of Al}}{7.780 \times 10^{-2} \text{ moles of alloy}} = 75.33 \% \text{ Al} \]

and

\[ \frac{1.919 \times 10^{-2} \text{ moles of Si}}{7.780 \times 10^{-2} \text{ moles of alloy}} = 24.67 \% \text{ Si} \]

Note that this is a hypereutectic Al-Si alloy.

As discussed in the chapter on microstructural evolution, the Si blocks attached to the \(\text{Al}_4C_1\) needles are thought to precipitate from the liquid hypereutectic Al-Si solution on cooling. The mass fraction of Si expected to form from a hypereutectic Al-Si alloy can be determined from tie-line calculations using the Al-Si phase diagram (the composition of the eutectic in this system is 12.2 at. \%):  

\[ \% \text{ Si} = \frac{(24.67 - 12.2) / (100 - 12.2) \times 100 = 14.20 \%} \]
and

\[ \% \text{ Al alloy} = \frac{(100-24.67)}{(100-12.2)} \times 100 = 85.80 \% . \]

Where the Al alloy is of the eutectic composition.

In terms of numbers of moles the alloy contains:

\[ 14.2 \% \text{ Si} \times 7.780 \times 10^{-2} \text{ moles of alloy} = 1.105 \times 10^{-2} \text{ moles of Si} \]

and

\[ 85.8 \% \text{ Al alloy} \times 7.780 \times 10^{-2} \text{ moles of alloy} = 6.675 \times 10^{-2} \text{ moles of Al alloy} \]

along with

\[ 4.547 \times 10^{-3} \text{ moles of Al}_4\text{C}_3 \]

These values must be converted to relative volumes to allow comparison with values of volume fraction determined from image analysis. The number of moles of each phase is multiplied by the ratio of the molecular weight to the density of each to find the volume. The volumes of each phase are:

\[ 4.547 \times 10^{-3} \text{ moles of Al}_4\text{C}_3 \times (143.959 \text{ gm/mole}) \times (1 \text{ cm}^3 / 2.990 \text{ gm}) = 0.2189 \text{ cm}^3 \text{ Al}_4\text{C}_3 \]

and

\[ 1.075 \times 10^{-2} \text{ moles of Si} \times (28.0855 \text{ gm/mole}) \times (1 \text{ cm}^3 / 2.330 \text{ gm}) = 0.1332 \text{ cm}^3 \text{ Si} \]

and

\[ 6.675 \times 10^{-2} \text{ moles of Al} \times (26.9815 \text{ gm/mole}) \times (1 \text{ cm}^3 / 2.657 \text{ gm}) = 0.6779 \text{ cm}^3 \text{ Al alloy}. \]
This gives a total of 1.0299 cm$^3$. Recall that the basis for this calculation was 1 cm$^3$ of Al-MMC. Hence there is a volume increase of approximately 3%. The volume fraction of each phase is:

$$0.2189 \text{ cm}^3 \text{ Al}_4\text{C}_3 / 1.0299 \text{ cm}^3 = 21.25 \text{ vol. \% Al}_4\text{C}_3$$

and

$$0.1414 \text{ cm}^3 \text{ Si} / 1.0299 \text{ cm}^3 = 12.93 \text{ vol. \% Si}$$

and

$$0.6707 \text{ cm}^3 \text{ Si} / 1.0310 \text{ cm}^3 = 65.82 \text{ vol. \% Al alloy.}$$

Note that the Al alloy also contains both Al (~87.8 wt. \%) and Si (~12.2 wt. \%). The results of the image analysis showed the microstructure to contain ~19.5 vol \% Al$_4$C$_3$, 10.5 \% blocky Si and 69.5 vol. \% Al alloy (21 vol.\% Al-Si eutectic and 48.5 vol \% \(\alpha\)-Al). While these values are not exactly the same as the values measured using image analysis, they are approximate and provide some corroboration to the model for microstructural evolution.

Errors are involved in both the measurements and calculations. The calculations rely on the accuracy of the measured Si and SiC contents of the as-received Al-MMC. Measurement of the volume fraction of the Si and Al$_4$C$_3$ was complicated by their similar gray color, and accurate measurement of the volume fraction of Si and Al in the eutectic was complicated by the fact that this system forms a divorced eutectic.