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Joining of aluminum-based particulate-reinforced metal-matrix composites

Kolli, Sudhakar, Ph.D.
The Ohio State University, 1990

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JOINING OF ALUMINUM BASED PARTICULATE-REINFORCED METAL-MATRIX COMPOSITES

DISSERTATION

Presented in partial fulfillment of the Requirements for
the Degree of Doctor of Philosophy in the Graduate
School of the Ohio State University

Sudhakar Kolli, B.E., M.Tech., M.S.

*********
The Ohio State University
1990

Dissertation Committee :
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D. G. Howden

Approved by

Advisor
Department Of Welding Engineering
To my dear parents
Dr. Kolli Pitchaiah and Mrs. Sakuntala Devi
for their wonderful love and affection
and
for their wise guidance and counseling
which gave unlimited opportunities to me.
ACKNOWLEDGEMENTS

I wish to express my sincere appreciation for my advisor Dr. D. W. Dickinson for his encouragement, understanding, guidance and counseling throughout the course of my dissertation work. I also want to thank my co-advisor, Dr. W. A. Baeslack III, for his encouragement, very helpful technical discussions and advice. I wish to record my appreciation for Dr. D. G. Howden for his suggestions. Dr. John Lippold of the Edison Welding Institute, Columbus gets my special praise for his suggestions, advice and encouragement. I want to thank Drs. J. E. Gould and Robert Rivett of the Edison Welding Institute for their useful suggestions in the beginning of the project. Financial support from the Edison Welding Institute, Columbus is gratefully acknowledged. I also express my appreciation for my sister Jyotsna whose encouragement was very inspiring for my work. Finally, I wish to place on record my special thanks for my wife Vijaya and children Sireesha and Venkat who have endured my very long working hours and without whose help and support this effort would not have been possible.
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Fields of Study

Major Field: Welding Engineering

Studies in

1. Physical Metallurgy
2. Welding Processes
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40. 25 Vol% SiCp/6061 Al MMC 3/8" diameter bars liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi. The resulting deformation at the interface is 41 %.

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( a ) Bond zone, note the flow lines disturbed near the interface ( at A ) and ( b ) a close up view suggesting particulate enrichment near the interface ( in the area B - B ).

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(a) shows a small void at the bond line (shown by arrows) and (b) at 15000 X shows the same. No particulate-matrix interfacial debonding may be noted;

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55. Chemical analysis of the region across the interface, by EPMA, of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 " silver interlayer, ten micron beam rastering, showing only aluminum and silver profiles (Note the difference in the scale for silver).

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61. Micro-hardness evaluation on a line normal to the interface (for 3.6 mm on either side of the interface) of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 " silver interlayer at 27.6 % deformation (B. M.: Base material).

62. Microhardness profile (for 3.75 mm on either side of the bond interface) for a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer, and post bond heat treated as per schedule # 1.
63. Fracture surface of a three point bend tested 25 Vol% SiCp/6061 Al MMC 3/8" diameter (as received) bar cut lengthwise;

(a) at low magnification shows dimples on fracture surface and (b) at a higher magnification shows particulates in addition to the dimples.

(c) view of dimpled fracture at high magnification. Note no interfacial debonding between the matrix and reinforcement.

64. Fracture surface of a 25 Vol% SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi, with a deformation at the interface of 27.6%, cut lengthwise and three point bend tested;

(a) at low magnification and (b) at a higher magnification. Note the reduction dimple size as compared to Figs. 47.

(c) at a high magnification. Note the dimples and reduction size of dimples as compared to Figs. 48. No interfacial debonding between the matrix and the reinforcement is observed.

65. Fracture surface of a 25 Vol% SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes with a linear deformation at the interface of 1%, (a) at 10000 X shows dimples and (b) at 20000 X shows an enlarged view of (a).

66. SEM fractographs of a 25 Vol% SiCp/6061 Al MMC base material;

(a) macroscopic fracture pattern at a low magnification and (b) fracture surface at a high magnification shows dimples, voids and particulate fracture (at arrow). Note no interfacial debonding;

(c) matching half of the fracture surface shown in Fig. 66 (b). Note the dimples, voids and particulate fracture (at arrows). No interfacial debonding between the matrix and the reinforcement is observed.

67. Fracture surface (tensile tested) of a 25 Vol% SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes with a deformation at the interface of 8.1%; (a) at low magnification
shows cracks and ( b ) at high magnification shows dimples and particulates. Note interfacial debonding at A.

68. Fracture surface (of a 25 Vol% SiCp/6061 Al MMC 3/8 " diameter bar liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 20 minutes with a deformation at the interface of 8.1 %; ( a ) shows void and ( b ) at high magnification shows an enlarged view shows dimples and particulates. Note no interfacial debonding;

( c ) the heavy particulate concentration may be noticed.

69. Fracture surface of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 20 minutes with a deformation at the interface of 10 %; ( a ) at 3000 X shows features of flat and dimpled fracture and ( b ) at higher magnification shows cracks and debonding; ( c ) shows dimples, voids and particulate fracture and ( d ) at higher magnification, shows second half of fracture surface with dimples, voids and particulate breakage ( at arrows );

( e ) fracture surface shows dimples and voids.

70. Optical macro and micrographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 10 minutes with a deformation at the interface of 12.6 %; a ) shows the bond region at a very low magnification. Note the misalignment between the bonded halves and ( b ) at higher magnification shows the large number of voids in the central region at the bond interface;

( c ) the bond region near the edge at a high magnification ( note voids ( A ) and particulate enrichment ( B ) at the interface ); ( d ) at a very high magnification shows an enlarged view of the bond near the edge. Note the particulate free region in both figures and its curvature ( C ) in ( d ) suggesting material flow towards the interface.

71. SEM fractographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 5 minutes with a deformation at the interface of 20.9 %; ( a ) shows a low magnification view of the central region of the fracture surface. Note voids ; ( b ) a high magnification view of
the above figure. Notice a large number of dimples, voids and a small number of particulates on the surface. Also note reinforcement - matrix interfacial debonding at A;

(c) shows a low magnification view of the edge region of the fracture surface; (d) a high magnification view of the above figure. Notice an extremely small number of dimples (as compared to (b)), voids, some flat fracture features and a very small number of particulates on the surface;

matching half of the surface shown in (a) to (d); (e) shows a low magnification view near the edge region of the fracture surface; (f) a high magnification view of the above figure. Notice a very limited number of dimples, flat fracture features (A) and voids on the surface.

72. SEM fractographs of a 25 Vol% SiC\textsubscript{p}/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 ° C for 5 minutes with a deformation at the interface of 10 %;

(a) shows a low magnification view near the edge region of the fracture surface; (b) a high magnification view of the above figure. Notice a very limited number of dimples, particulate fracture (at arrow), reinforcement - matrix interfacial debonding (A) and voids on the fracture surface;

(c) shows a low magnification view near the central region of the fracture surface; (d) a high magnification view of the above figure. Notice a very limited number of dimples, flat fracture features (A), particulate fracture (at arrow) and voids on the surface.

73. Fracture surface of SiC\textsubscript{p}/6061 Al tensile tested at 900 F (Ref. 115). Features of fibrous fracture similar to those observed in Fig. 30 (b) may be noticed.

74. Results of Gleeble thermo - mechanical simulation for a simulation time of 30 minutes.

75. Summary of the results of base metal thermo - mechanical simulation.

76. Summary of recommended bonding parameters zone based on the thermo - mechanical simulation studies.

77. Macrograph of a 25 Vol% SiC\textsubscript{p}/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 ° C for 20 minutes with a deformation at the interface of 7.8 %. The sample was additionally held at 500 ° C for 40 minutes.
Macro and Micrographs of a 25 Vol% SiCp/6061 Al MMC, liquid-phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 60 minutes with a deformation at the interface of 10%; (a) macrograph showing extensive cracking transverse to the interface and (b) shows large voids on the bond line and a crack close to the bond line at a low magnification.
CHAPTER I

1.0.0 INTRODUCTION

Developments in aerospace material technology are taking place at an accelerated pace, with the rapid progress in materials science making a significant contribution. Considering the traditional gas-turbine engine industry for aeronautical applications, continuous improvements in the turbine inlet temperatures are being achieved through application of new and engineered materials. The goals of the aerospace industry have been to increase the specific load carrying capacity and specific fuel consumption. This drive for structural weight reductions applies to all major areas from the automobiles to space vehicles. This is being accomplished, partly, through the development of new materials. It is in this context that metal matrix composites are being considered as possible structural materials to replace the monolithic metallic alloys. Metal matrix composites (MMCs) are finding increasing prospects for weight-critical applications in aerospace, automotive, sports and opto-electronic instrumentation\(^1\) applications. Their attractiveness for structural applications stems from their high specific strength and high specific stiffness as compared to conventional metallic alloys. They are generally available in the following forms:
i) reinforcement by continuous fibers of a high strength material

ii) reinforcement by whiskers or short fibers of a high strength material

iii) reinforcement by particulates or equi-axed particles of a high strength material.

Reinforcements for composite materials range from silicon carbide to alumina and graphite materials\(^2\). The continuous fiber reinforced composites have higher specific strength in the fiber direction and better fracture toughness than the whisker or particulate reinforced composites. The particulate reinforced composites have much in common\(^2\) with precipitation/dispersoid strengthened materials excluding the problems such as precipitate/dispersoid coarsening. The whisker (w) and particulate (p) reinforced composites can be fabricated by the same routes as conventional metallic materials, such as extrusion, rolling, forging\(^3\) and machining. They also exhibit isotropic properties. Thus, they show great promise for stiff and light-weight structural applications requiring reasonable corrosion resistance\(^2\).

Typically the reinforcements used in the composite materials are silicon carbide, boron, titanium carbide, alumina, graphite, titanium boride etc. Among the reinforcement materials, silicon carbide (SiC) has advantages of excellent thermal conductivity, good corrosion resistance and a comparatively low whisker cost versus boron, graphite and alumina\(^2\). In addition, SiC is also chemically compatible with aluminum and bonds with the matrix without forming intermetallic phases\(^2\). The aluminum alloys which have been produced as MMC's include 2024, 6061, 7075, 7090, 7091 and 8090.
Joining and other processes such as machining and finishing are treated as secondary fabrication processes whereas the composite material manufacturing is referred to as the primary fabrication process. The potential for the application of MMC's for structural fabrication will be fully realized only when appropriate methods for joining are identified and successfully applied with minimal damage to the engineered properties. The process of joining MMC's resembles somewhat the joining of other engineered materials such as rapidly solidified materials in that the need to preserve the as-manufactured engineered structure and properties becomes very critical.

Among joining methods, arc welding is one of the most versatile and widely applied methods for joining monolithic metallic alloys. The acceptance of fusion welding as a viable joining process requires the preservation of reinforcement - matrix interfacial integrity and properties and the attainment of a mechanically and metallurgically sound weld. In the joining of MMC's arc welding is not widely used, in view of the potential for damaging the engineered properties. Even high energy density welding processes such as electron beam and laser welding should be evaluated with the above objectives in spite of their potential for minimal damage to properties due to the low heat input attainable with these methods relative to arc welding. Solid - state joining techniques, such as inertia welding, diffusion welding, capacitor discharge welding and liquid phase diffusion bonding, have the potential for producing joints in MMC's with properties approaching that of the as - fabricated materials.

In chapter two, the composite materials primary processing is briefly introduced and the physical metallurgy and mechanical properties are outlined with particular emphasis on SiCp/6061 Al MMC. In addition, it also reviews the joining methods ( both
fusion and non-fusion) previously used with their relative advantages and disadvantages. Among the solid state techniques, diffusion bonding is reviewed with more emphasis placed on the liquid phase diffusion bonding. After careful evaluation, the liquid phase diffusion bonding method was chosen in this study to join 25 Vol % SiCp (silicon carbide particulate) /6061 Al MMC. Certain aspects of this process such as interlayer materials, modeling of isothermal solidification etc. will be also analyzed in the second chapter.

Chapter three outlines the objectives of the study. In this work, the thermal and thermo-mechanical simulation of the base material was performed to identify the effects of process variables such as time, temperature and mechanical loading on the joining of the composite material. Based on this analysis, a window of appropriate joining conditions was developed. The analysis steps include the characterization of microstructures with optical and scanning electron microscopy and the evaluation of mechanical properties.

In chapter four the experimental methods are discussed. The experimental details regarding thermal simulation using conventional furnaces and thermo-mechanical simulation with the Gleeble 1500 system are provided. In addition, the sample designs, equipment and methods for simulation as well as bonding are presented. Subsequently, the methods of surface cleaning, metallographic preparation and the microstructural characterization of the simulated/bonded samples are discussed. Chapter four also includes the details of the techniques of mechanical property evaluation for both the simulated and the bonded samples.
In chapter five, the results of the experimentation are presented. Initially the results of base material heat treatment and phase transformation were provided. This provides information regarding the heat treatment of the 25 Vol % SiCp/6061 Al MMC. Some base material samples were solution treated, naturally aged and then artificially aged for different times. The variation of microhardness with the artificial age - hardening time is recorded. This would generate information for the heat treatment of the bonded samples. Though, composite processing is not expected to alter the solidus and the liquidus temperatures of the 6061 Al matrix alloy, it was decided that differential scanning calorimetric studies would be performed to confirm this.

Subsequently the results of both the thermal and thermo-mechanical simulation are provided. They include the details of mechanical properties such as microhardness and tensile strength and microstructural investigations of metallographically polished surfaces with optical microscopy and of fracture surfaces with the scanning electron microscope. Based on the details from the mechanical property and microstructural investigations as above, a window of appropriate bonding conditions was developed. The basic idea in providing this window was to identify and minimize conditions that would otherwise degrade the base metal mechanical properties and microstructure. At this point, the process parameters to be used for the experimentation were developed.

The next sections of chapter five provide the experimental results of the liquid phase diffusion bonding and discussions. Initially, the results of bonding of 6061 aluminum alloy with an Al-Si-Mg ternary interlayer are presented and discussed. The computation of time for isothermal solidification with the above interlayer is also discussed. Details of liquid phase diffusion bonding of 6061 Al bar stock with a 0.001 "
silver interlayer in butt joint configuration are discussed. Subsequently, experimental results of a 25 Vol % SiCp/6061 Al MMC bar stock, liquid phase bonded in a butt joint configuration with a silver interlayer of 0.001 " thickness, are presented. The microstructural investigations include details of optical and SEM microstructure. Initially the optical macro microstructures of the bonded samples are presented. Subsequently results of the electron microscopic (SEM) investigations of the bonded samples are provided.

The next part in chapter five gives results from the chemical analysis of the regions near the bond interface to confirm the diffusional processes. Considering that initially a commercially pure interlayer was present at the interface prior to bonding, chemical analysis after bonding of the same area would reveal if any part of the original interlayer remained at the interface. It also provides information about the nature and extent of the diffusional processes occurring at the interface. Electron probe microanalysis (EPMA) and energy dispersive x-ray microanalysis (EDAX) studies on the SEM provided this information. The results of the probe size variation on the EPMA data are also included. After obtaining the above information, which confirms the presence of the original silver remaining at the interface, if any, the next is to assure that the liquid phase occurred at the interface. Here, the results obtained by utilizing a thicker silver interlayer (0.005 " or 0.127 mm) are outlined. The details of the optical microscopic investigations and the chemical analysis data from the electron microprobe are presented.

Mechanical properties of bonded joints, such as tensile strength and microhardness and fractographs of bend and tensile tested samples are subsequently
presented and discussed. SEM fractographs of the fracture surfaces of the heat treated base material and the bonded samples are next provided. Some additional results including optical macro and micrographs and SEM fractographs are subsequently provided.

The discussion chapter (Chapter VI) summarizes the observations of the experimental results. Here the results of the base material studies such as heat treatment, phase transformation and thermal and thermo-mechanical simulation are discussed and necessary bonding parameter zones are highlighted. The results of the bonds with the Al-Si-MG interlayer are analyzed and the microstructures of the MMC bonds with the silver interlayer are discussed. Subsequently the confirmation of liquid phase bonding is discussed. The fracture surfaces of the tension tested bonds including those of the additional experiments (taken up to investigate the effects of shorter bonding temperatures and times) are analyzed. Chapter seven outlines the conclusions and a list of references is provided at the end.
CHAPTER II

2.0.0 BACKGROUND

2.1.0 PROCESSING OF METAL MATRIX COMPOSITES

Metal matrix composites are produced by various techniques involving solid state, solid-liquid and liquid phase techniques. The latter process involves the liquid phase processing of the matrix material, with a solid state reinforcement.

2.1.1 REINFORCEMENT MATERIALS

The reinforcements are made from silicon carbide, graphite, alumina, boron, boron carbide, titanium boride and other materials. Traditionally, the reinforcement materials have been expensive and this contributed to the slow progress in non-aerospace applications of metal matrix composites. Continuous SiC filament is produced by Chemical Vapor Deposition. A pyrolitic graphite coated carbon monofilament substrate is exposed to silane and hydrogen gases. Silane decomposes to form beta silicon carbide on the substrate. With the above process fibers of 5.6 mils diameter can be produced.
The CVD SiC fiber has an average tensile strength of 550-600 ksi and an elastic modulus of 58-60 msi. The β-SiC whiskers made from pyrolyzing rice hulls, which have been available since 1973, are cheap. The whisker has a cubic crystal structure. SiC is less costly, bonds well to metals and resists strength degradation under elevated temperature processing, better than boron. SiC whiskers (SiC\textsubscript{w}) have a tensile strength approaching 1000ksi\textsuperscript{5}. An analysis of the the bulk chemistry of the SiC whiskers shows a carbon level close to the theoretical value (30%)\textsuperscript{6}. Oxygen contents were observed to be around 0.92 wt%. The surfaces of the SiC whiskers can have SiO\textsubscript{2} or Si-O-C glass depending on processing route, surface impurities and surface oxygen content. Whiskers range from 20 nm to 0.5\mu m in diameter and can be up to 100 \mu m in length\textsuperscript{6}. Morphologically, the whiskers can be straight, bent or branched. The straight whiskers constitute the bulk of reinforcement in whisker-reinforced materials. In addition there can be whisker or particulate debris present.

The attainment of optimum properties in the composite depends on the features of the reinforcement. The important factors, in case of particulate reinforced material, include: particle morphology, particle size distribution, particle surface chemistry, fractional content and dispersion uniformity\textsuperscript{1}.  

2.1.2 MATRIX MATERIALS

The matrix materials used for metal matrix composites include aluminum alloys 2024, 2124, 5156, 6061, 7075, 7090, 7091, 8090, magnesium alloys, titanium alloys, copper and others\textsuperscript{2,7}. The basic requirements and purposes of the matrix materials are to :
i) provide ductility;

ii) transfer load between individual reinforcement materials;

iii) in case of fiber reinforced materials, provide transverse strength.

2.1.3 SOLID STATE PROCESSING

The primary processing of composite materials by the powder metallurgy route involves the following steps.

i) Pre-alloyed metallic powders are mixed with silicon carbide powders or whiskers and blended, taking care to avoid explosions due to the fine metallic powder

ii) The resulting mix is cold compacted and outgassed. Heating at a low temperature (150°C) under vacuum helps desorb the volatile contaminants. Further outgassing may be necessary based on the powder composite system being employed.

iii) Consolidation is achieved through vacuum hot pressing. Hot pressing temperature may be above the solidus of the alloy. The temperatures and times are generally considered proprietary. Uniformity of temperature and time at temperature necessary for equilibration should not be underemphasized. Generally the high consolidation temperatures and times required for a powder route processed composite result in formation of an interfacial zone.

iv) The billets produced through the above operations are extruded to necessary shapes and sizes. Extrusion also helps in fragmenting the prior
oxide skins at the reinforcement surfaces. Through the above processing it is possible to achieve a uniform distribution of particulate/whisker reinforcement. In the extrusion process, many of the silicon carbide particles can be fractured and thus the size distribution of particles can be very different from that in the starting stage.

Hot isostatic pressing, though widely utilized for conventional metallic alloys, is not used extensively for the fabrication of aluminum based composite materials.

2.1.4 LIQUID METAL INFILTRATION

Liquid metal infiltration is a second method for producing metal matrix composites. This process relies on the injection and subsequent solidification of liquid metal into the interstitial spaces within the framework of a ceramic reinforcement. The forms of liquid metal infiltration include squeeze casting, pressure casting, die casting, gravity casting and centrifugal casting. The sequence of operations include the introduction of reinforcement material into the melt of the selected matrix material and subsequent solidification of the melt under desired conditions. In addition, careful analysis and control of wetting kinetics between the reinforcement and the matrix material are very crucial. The parameters of importance for wettability include, heat of formation of the oxides at the interface, valence electron concentration, interfacial reactions, surface geometry, temperature and time. Mixing of reinforcement with molten alloys or pressure infiltration of the latter into ceramic preforms helps achieve intimate contact and bonding subject to wettability. Thus, with the casting process, MMCs with a tailored set of engineering properties, not achievable with conventional monolithic materials, can be
produced. The melt infiltrated particulate/whisker reinforced composites can also be processed by conventional metal working technology, like their powder metallurgy fabricated counterparts.

Techniques such as sand casting can give rise to buoyancy-driven segregation of particles whereas a more homogeneous distribution can be attained in die casting. A particle rich zone or a particle impoverished zone can be found in centrifugal castings based on the density of the reinforcement. Compocasting relies on the incorporation of the reinforcement particulates or discontinuous fibers into a vigorously agitated melt of the matrix slurry held between the liquidus and the solidus temperatures. Chemical reactions at the interface aided by an increase in time can improve wettability.

Squeeze casting or liquid forging produces void-free castings through pressurized liquid metal infiltration of ceramic preforms. This process provides an equi-axed grain structure resulting from large undercoolings and rapid heat extraction.

Other techniques such as vacuum infiltration and investment casting have also been explored. In all of the above techniques, fluidity, as dictated by the variations in melt viscosity due to additions of ceramic reinforcements to the matrix alloy melt, also plays an important role in achieving the uniform dispersal of the reinforcement in the casting.
2.1.5 OTHER TECHNIQUES

Rapid solidification 16, spray deposition 17, in situ fabrication 18 (liquid metal techniques), electrodeposition and plasma-spray deposition 19, roll diffusion bonding 20 (solid state techniques), and laser beam processing 21 (solid-liquid technique) are some of the other routes used in the production of composite materials. It is claimed that melt infiltration offers better bonds between SiC and the aluminum matrix than the powder metallurgy route through provision of "prior wetting contact" with the liquid phase 16. However, it is not clear how the liquid phase alone can provide good "prior wetting contact". In the spray deposition process, inert gas atomized molten metal droplets are collected along with the ceramic particles on a ceramic substrate. It is observed that there is very little precipitation at the SiC interface in this process as compared to the powder metallurgy route 17. In situ fabrication relies on the "Controlled Unidirectional Solidification" of a eutectic alloy. Electrodeposition and plasma-spray deposition are among the earlier methods used to fabricate composites with reactive filaments such as titanium.

2.2.0 PHYSICAL METALLURGY AND MECHANICAL PROPERTIES

The engineering properties of metal matrix composites depend on their constituents, their physical forms, chemical reactivities, interfacial and microstructural properties, and production processes. The subsequent information will be largely restricted to SiC-reinforced aluminum metal matrix composites.
2.2.1 PHYSICAL METALLURGY

The information required to characterize interfaces in MMCs can be quite exhaustive. Geometry and dimensions, microstructure and morphology, mechanical, physical, chemical and thermal characteristics of different phases are only some of the many aspects. Since metal matrix composites are non-equilibrium systems, a chemical potential gradient exists across the reinforcement-matrix interface. This results in the formation of an interfacial interlayer with chemistry and properties different from either that of the matrix or the reinforcement. The nature, extent and crystallinity or the lack of crystallinity of this interlayer, depends on the chemistry and reactivity of the constituents and their thermo-mechanical processing history. The local chemistry of the interface can affect the bonding at the interface (bonding at the interface can be categorized as mechanical and chemical (dissolution, wettability and reaction)).

This in turn can have an effect on the mechanical properties and the corrosion resistance of the composite since it serves to transfer load between the lower modulus matrix and the higher modulus reinforcement. In addition, the interface itself is a region wherein discontinuity of properties such as differences in coefficients of thermal expansion exists, resulting in residual stress.

Considering the powder metallurgy route, it is claimed that bonding between silicon carbide and the aluminum matrix is quite strong, as evidenced by fracture analysis of tensile specimen. A strong aluminum-SiC interface is also reported based on auger analysis of in-situ fractured material. Flom and Arsenault determined the interfacial bond strength of an aluminum alloy (6061) and SiC to be 1690 Mpa (244.9 ksi), which is 40 times higher than the yield stress of the annealed
aluminum alloy. This was based on an experimental technique to determine local stresses during plastic deformation under a triaxial stress state. They used a 1Vol% SiC reinforced 6061 aluminum to satisfy the criteria for non-interacting particles and assumed the particulates to be spherical. But, in reality the reinforcement level will be much higher and the particulate may not be spherical. Also, it has been observed that extrusion fractures SiC particles, producing a particulate size distribution. Floma's theoretical analysis was based on an an earlier work by Argon et al. The good bonding strength between SiC and aluminum has been attributed to substantial dislocation generation (a density of 1 to 4 x 10^{10} / cm^2) at the interface.

Precipitates have been observed at the interfacial region in 7xxx and 2124 Al/SiC composites. Intermetallic compounds, dispersoids and preferential magnesium enrichment have also been observed at the interface. In a 7xxx Al / whisker (SiCw) composite, MgO crystallites have been detected at the reinforcement - matrix interface. In a SiC/6061 Al composite, a 30 angstrom thick layer of MgO has been reported, at the interface. Thus, it appears that in powder metallurgy processed materials oxides are incorporated due to the prior surface oxide films on powder particles. It is possible that the formation of the more stable MgO has occurred through reduction of aluminum oxide during the elevated temperature processing required for the consolidation of the composite. In addition, super-solidus processing seems to have contributed to the formation of intermetallic compounds at the matrix - reinforcement interface. The nature and morphology of oxides, intermetallic compounds and precipitates, in addition to the different phases present at the interface, affects the mechanical properties and corrosion resistance of the composite.
Optical microstructure of the SiC/6061, 2014, 2124 Al composites shows the whisker/particulate material aligned in the extrusion direction, reinforcement free zones and whiskers/particulates of various sizes. The size distribution may be caused by reinforcement breakages during extrusion. It is almost impossible to observe the grain structure of the matrix due to the reinforcement material. Transmission electron microscopy shows subgrain formation and a high dislocation density in the matrix and at the particle/matrix interface. This high dislocation density in the matrix seems to influence microstructural development, aging behavior and strength. It is postulated that the relatively high difference in thermal expansion coefficients between aluminum and SiC contributes to the large dislocation density in the matrix. In addition, extremely fine precipitates have been observed in the 6061 matrix and along the dislocations.

Due to the lengthy elevated temperature consolidation of MMC's, an interfacial zone is formed at the reinforcement matrix interface. Nutt, utilizing transmission electron microscopy (TEM), investigated the interfaces in 6061 Al matrix composites reinforced with SiC whiskers and observed that the whisker-matrix interfaces were "often characterized by a continuous layer of polycrystalline oxide particles". Compositional analyses confirmed the presence of Magnesium and oxygen and the "lattice spacings within the oxide layer were consistent with MgO (200) planes". Fig 1 (Ref. 29) shows a picture obtained by Nutt. The oxide layer, which was 3 nm thick, was observed to be distinct and separate from both the matrix and the reinforcement. It is possible that the interface layer was developed during the elevated temperature vacuum hot pressing required by the powder metallurgy route processed composite. SiC reinforcements usually have SiO2 or Si-O-C coatings. During the
Fig. 1 A continuous layer of polycrystalline oxide (MgO), 3nm thick, along the SiC whisker - 6061 Al matrix interface (Ref. 29).
semi-solid processing, magnesium from 6061 Al matrix, has probably reduced the SiO₂ and formed the crystalline MgO. If the MgO coating is continuous, as reported by Nutt, then this could restrict further interaction between the aluminum rich liquid and SiC during the semi-solid processing. However, Fu et al. 34, investigated a 25 Vol% SiCp/6061 Al by TEM and observed no interface phases. They suggested that the interfacial region could be too thin to be observed in the limited number of specimen tested by them or could be amorphous. In the case of short fiber reinforced SiC/6061 Al obtained from M/S Arco Chemicals, Fu et al. 34, observed a γ-Al₂O₃ phase at the reinforcement-matrix interfaces. Nutt's 29 whisker reinforced composite material was obtained from Arco Chemicals, Greer, South Carolina whereas Fu's 34, particulate reinforced composite material was obtained from DWA composites, California. The differences in the sources of origin of material could mean differences in their reinforcement suppliers and differences in their manufacturing procedures (Vacuum pressing temperatures and times), which may influence the interface phases. Divecha et al. 3, 23, Flom et al. 25 and Fu et al. 34, confirmed that the interfacial bonding in SiC/6061 Al was quite strong. Based on the above information, it is expected that a continuous layer of MgO may be present in the SiCp/6061 Al MMC, procured for the current study from Advanced Composite Materials Corporation, Greer, South Carolina. It may be noted that Nutt's 29 whisker reinforced material was also manufactured by the same corporation.

The heat treatment of SiC reinforced 6061 Al composite differs from that of the matrix alloy, although the basic steps, i.e., solution treatment and age hardening, are the same. The age hardening sequence in SiCₜ₉ reinforced 6061 aluminum alloys 35 is essentially that reported for ingot metallurgy 6061 Al alloys 36, 37, i.e., supersaturated
solid solution - vacancy-silicon clusters - vacancy rich coherent Al-Mg-Si GP Zones - disordered partially coherent $\langle 100 \rangle_{\text{Al}}$ needle-shaped phase ($\beta^\prime$) - ordered, partially coherent $\langle 100 \rangle_{\text{Al}}$ needle-shaped phase ($\beta^\prime$) - semi-coherent, hexagonal rod-shaped phase - equilibrium $\beta$-Mg$_2$Si platelets. The differences between 6061 Al and SiC reinforced 6061 Al, with regard to age hardening, are the following:

i) SiC reinforcement makes 6061 Al quench sensitive (differences in age hardening behavior due to variations in the quenching rate have been observed);

ii) the high dislocation density in the composite restricts the number of GP zones formed through a reduction in the vacancy concentration and accelerates the formation of $\beta^\prime$ and $\beta^\prime$;

iii) the formation of $\beta^\prime$ is more accelerated compared to that of $\beta^\prime$.

Inhibition of GP zone formation has also been reported in alumina/6061 Al composite. However Rack presents a transmission-electron micrograph for a 20 Vol% SiC$_w$/6061 Al solution treated at 538 °C, water quenched and direct aged at 155 °C for 10 hours which shows virtually no $\beta^\prime$, $\beta^\prime$ and $\beta$ precipitates. He suggests that in the case of age hardening at low temperatures, the dislocation substructure may not greatly benefit age hardening while increased dislocation density helps promote the transition phases while age hardening at higher temperatures.

Summarizing the above, the heat treatment behavior of SiC/6061 Al composite materials differs from that of ingot metallurgy 6061 Al or that of powder metallurgy route processed 6061 Al due to the substantiative dislocation generation during the material
processing arising from the thermal expansion differences between the matrix and the reinforcement. This alters the aging kinetics with the GP zone formation being inhibited and the formation of $\beta'$ and $\beta''$ being accelerated. This results in the peak hardness being obtained at earlier times than in the conventional 6061 Al alloys\(^{25, 27, 36, 38}\). In addition, a step of natural aging is said to reduce the aging time required to achieve the maximum hardness\(^{35}\).

2.2.2 MECHANICAL PROPERTIES

Table 1 shows typical mechanical properties of SiC\(_p\) / SiC\(_w\) reinforced 6061 aluminum alloy composites. This data refers to powder metallurgy route processed composites. From the data presented in the table it may be observed that the addition of 25 Vol\% Silicon Carbide particulate (SiC\(_p\)) reinforcement to 6061 aluminum increases the elastic modulus by 65\%, tensile strength by 66.67\% and causes a reduction in ductility from 12 to 4.5\%. Though there is no specific reference to the heat treatment condition for the SiC\(_p\) reinforced composite data from Harrigan\(^7\), we can assume that the those values represent a T-6 condition. Data from Logsdon and Liaw\(^{39}\), contrary to the data from Harrigan, indicates that the room temperature yield, tensile strength and total elongation of SiC\(_p\)/6061 Al are slightly better than that of SiC\(_w\)/6061 Al. The ductility of SiC reinforced 6061 Al composites can be improved substantially by hot rolling\(^{40}\). Mechanical working is also reported to improve ductility in SiC/Al composites\(^{41}\). For discontinuous reinforcements of SiC in aluminum, the modulus is observed to be isotropic and independent of the type of reinforcement or the matrix alloy\(^{41}\). The literature also indicates that, compared to the matrices of 2024, 2124, 5083 and 7075 Al, the 6061 Al matrix appears to possess "good strength and higher ductility", with a 20
Table. 1  Typical mechanical properties of SiC particulate/whisker reinforced aluminum alloy composites.

<table>
<thead>
<tr>
<th>Material : 6061 Al or 6061 Al MMC</th>
<th>Modulus</th>
<th>Yield Strength</th>
<th>Ultimate tensile strength</th>
<th>Ductility</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wrought 6061 Al T-6</td>
<td>10</td>
<td>40</td>
<td>45</td>
<td>12</td>
</tr>
<tr>
<td>25 Vol % SiCp Al 6061 (Ref. 7)</td>
<td>16.5</td>
<td>62</td>
<td>75</td>
<td>4.5</td>
</tr>
<tr>
<td>25 Vol % SiCp Al 6061T-6 (Ref. 39)</td>
<td>***</td>
<td>59.1 T</td>
<td>68.1 T</td>
<td>2.0 T</td>
</tr>
<tr>
<td></td>
<td></td>
<td>61.7 L</td>
<td>70.9 L</td>
<td>1.8 L</td>
</tr>
<tr>
<td>6061T-6 (Ref. 39)</td>
<td>***</td>
<td>53.9 T</td>
<td>64.3 T</td>
<td>1.9 T</td>
</tr>
<tr>
<td></td>
<td></td>
<td>55.3 L</td>
<td>61.4 L</td>
<td>0.7 L</td>
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<tr>
<td>20 Vol % SiCw Al 6061T-6 Rod (Ref.1)</td>
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<td>18</td>
<td>68</td>
<td>87</td>
<td>2.8</td>
</tr>
</tbody>
</table>

*** : No information about modulus  
T : Transverse  
L : Longitudinal
Vol% SiC\textsubscript{w} reinforcement, T-6 condition (excluding 5083) and testing in the longitudinal direction \textsuperscript{41}. Heat treatment has little effect on the elastic modulus, but \textsuperscript{41} "steepens the slope of the stress-strain curve at the inception of plastic flow". Yield and tensile strengths of the SiC/Al composites, are controlled by the type of the matrix alloy for the same reinforcement type and level. SiC/6061Al achieved the highest strength relative to the matrix alloy compared to 2124, 5083 and 7075 Al matrices \textsuperscript{41}, and for the case of 20 Vol% whisker or particulate reinforcement in 6061 Al, there was no appreciable difference in strength. The strengthening of liquid metal infiltrated (melted) composites differs from that of the PM composites \textsuperscript{7}. The main problem in the former case arises from the segregation of reinforcement, a larger particulate size and the need for coating the reinforcement (resulting in lack of bonding \textsuperscript{42}) in the case of melted composites. Thus, though previous literature \textsuperscript{5,7} suggested that composites processed through liquid metal infiltration have inferior properties; Arsenault \textsuperscript{42} suggested that, there should be no differences in the strength levels between the two routes of processing if the reinforcement size, volume fraction and distribution remained the same.

The fracture toughness properties of aluminum metal - matrix composites are dependent upon both the matrix and the reinforcement materials. SiC reinforced 6061 Al composites are isotropic in terms of fracture toughness and strength, whereas SiC reinforced 2124 Al composites \textsuperscript{39} display a "fracture toughness-orientation dependence". Among them, SiC\textsubscript{p}/6061 Al has superior fracture toughness compared to SiC\textsubscript{w}/6061 Al. Logsdon and Liaw \textsuperscript{39} report that the fracture toughness of SiC\textsubscript{p} and SiC\textsubscript{w} reinforced aluminum metal matrix composites is about a third of the corresponding wrought aluminum alloys.
The interfacial region has a major role to play in the toughness of the composites. In 7xxx/SiC aluminum composites, at equal strength levels, low toughness has been noticed in overaged material with interfacial precipitation, as opposed to relatively higher toughness in the under-aged material devoid of any interfacial precipitates. T-6 Heat treatment of SiC/6061 Al has an adverse effect on fracture toughness and the resistance to fatigue crack propagation. In general, the effects of the addition of discontinuous reinforcement to Al on the fatigue crack growth resistance depend on the reinforcement size distribution, the stress intensity range, and the matrix material, but do not degrade the composites as compared to the monolithic materials near the threshold stress intensity range. Among SiCw/2124 Al, SiCw/6061 Al and SiCp/6061 Al, the last and the first exhibit superior and relatively inferior, fatigue crack growth resistance, respectively. SiCp/6061Al (F) exhibited the highest threshold stress intensity range (ΔKth) while SiCw/2124 Al exhibited the lowest ΔKth.

In summary, SiC whisker or particulate reinforcement degrades the fracture toughness somewhat. SiC particulate reinforcement in 6061 Al offers better fracture toughness properties than the whisker reinforcement in either 6061 or 2124 Al matrices. The SiC particulate or whisker reinforcement does not degrade the fatigue crack growth resistance in the 6061 aluminum alloys.

Creep studies of SiCw/6061 Al indicate very short primary and tertiary stages with a long period of steady state creep and a stress exponent of 20.5. This contrasts with the absence of a primary stage and a stress exponent of 2 to 3 in the creep of 6061 Al. The addition of SiCw, apparently, increases the creep resistance of 6061 Al, depending on applied stress and creep rate. The creep resistance of SiC whisker
reinforced 6061 Al is 100 times more than that of the particulate reinforced 6061 Al alloy 26.

In summary, particulate or whisker reinforcements offer isotropic mechanical properties, with the addition of SiC reinforcement to aluminum improves the elastic modulus, yield and tensile strength and causes a reduction in ductility and fracture toughness. Ductility can be improved by mechanical working. Addition of SiC reinforcement to 6061 Al matrix improves mechanical properties in a superior fashion compared to the reinforcement addition in 2124, 5083 and 7075 Al matrices. SiC_p/6061 Al displays superior fatigue crack growth resistance compared to SiC_w/6061 Al or SiC_w/2124 Al. SiC_p reinforcement offers similar yield & tensile strength, modulus, relatively superior fracture toughness and inferior creep resistance than SiC_w reinforcement in 6061 Al.

2.3.0. JOINING

The successful development of metal - matrix composites for advanced structural applications depends, principally on their ability to be joined to themselves and to other monolithic materials with the available conventional and advanced welding techniques. As already discussed in the previous section, MMC's consist of dissimilar materials, often with an interphase interface, which could be different from either the matrix or the reinforcement. Welding processes rely on the application of heat and or pressure. The thermal cycle, which, a material experiences in welding, depends on its physical properties and the process variables. The metallurgical changes in the material, due to the welding thermo-mechanical cycle, depend on its chemistry and physical metallurgy.
Thus, before a decision on the selection or application of a joining process is made for composites several factors need to be considered. There could be several difficulties due to the selection or application of an inappropriate process, such as:

i) degradation in the joint mechanical properties such as yield and tensile strength or ductility and a reduction in fracture toughness and fatigue limit compared to the base metal;
ii) porosity, delamination or fiber pull-out;
iii) destruction of the matrix reinforcement interface;
iv) undesirable chemical reactions leading to products which could worsen the material's corrosion resistance.

Some of the various joining techniques used for metal matrix composites are listed below:

i) Arc welding
ii) Laser beam welding
iii) Resistance welding
iv) Soldering and Brazing
v) Capacitor discharge welding
vi) Inertia welding
vii) Diffusion bonding

The literature information available on each of the processes is summarized below.
2.3.1 **ARC WELDING**

Gas tungsten (GTA) and Gas metal arc (GMA) welding processes were used to join Boron-Aluminum^45^, Titanium-Graphite^46^, Aluminum-Graphite^47^ and SiCw or SiCp-6061 Al^48^,^49^ composite materials. From these studies, it is clear that arc welding develops many problems encountered in joining composite materials, as listed below:

i) porosity;
ii) delamination and splitting of fibers;
iii) formation of undesirable chemical reaction products;
iv) degradation of mechanical properties;
v) cracking.

For example, in continuous fiber boron-aluminum, porosity, fiber dissolution and cracking were observed^45^. In the titanium-graphite and aluminum-graphite systems, titanium carbide and aluminum carbide layers, respectively, were observed after arc welding^46,47^. For the SiC/Al, a lower weld metal hardness compared to the parent metal or heat-affected zone hardness, porosity and formation of Al_{4}C_{3} in the fusion zone^23,48^ have been reported. Also, the yield strength values ranged from 60% for 20 Vol% SiCw/6061 Al tube welded (GMA) with 5356 filler material in the heat treated (T6) condition to 63% for 20 Vol% SiCw/6061 Al sheet, 0.125" thick, welded (GMA) with 5356 filler material of parent material strength^48^ (yield strength 40.9 Ksi). The ultimate tensile strength varied as a function of gap thickness, with the maximum being achieved at a gap of 0.04" (1.0 mm) dropping off sharply at smaller gaps due to difficulties in obtaining adequate fusion between weld and base metals. A lower strength...
has been reported for the GTA welding process as compared to the GMA process. The tensile strength for the latter is reported to be 255 Mpa (36.59 Ksi) in the as-welded and heat-treated condition compared to a base metal strength of 299 Mpa (42.9 Ksi), assuming the as-fabricated condition. Assuming a heat-treated base metal tensile strength of 578 Mpa (82.93 Ksi), this equals a joint efficiency of 44%. In the fusion joining of SiC/Al, the formation of Al₄C₃ should be considered critically, since it can endanger the engineered properties through the formation of corrosion. It is reported that in the presence of moisture, aluminum carbide decomposes with release of hydrocarbon gases." The reaction equation is given by:

\[
3 \text{SiC (s)} + 4 \text{Al (l)} \rightarrow \text{Al}_4\text{C}_3 + 3 \text{Si (s)} \quad (1)
\]

The aluminum carbide forming at the Al-SiC interface may interfere with stress transfer between the matrix and reinforcement. From thermodynamic considerations, it is not possible for the above reaction to proceed forward at temperatures below 730 °C. Since it is very likely that the temperatures will be much higher than this in arc welding, formation of aluminum carbide can be considered a certainty.

2.3.2 LASER BEAM WELDING

The laser welding process is capable of producing low heat input, high energy density welds. It is possible to attain a rapidly solidified weld with this process. However, very little information is available from the literature regarding the laser welding of composites. Develetian, in a study on joining of 40 Vol% SiCₚ/6061 Al, reports massive quantities of aluminum carbide and blocky silicon. This is to be
expected, since the temperatures reached in laser welds are likely to be such that a reaction between aluminum and silicon carbide occurs.

Also, the poor absorptivity of the CO\textsubscript{2} laser power by aluminum is another hindrance. It has been reported that an absorptivity of 12% , which is 4 times larger than that of monolithic aluminum, has been attained in laser processing of SiC/Al composite for primary fabrication \textsuperscript{21}. At a power density of 1.25X10\textsuperscript{7} J/m\textsuperscript{2}, severe reaction between matrix and the reinforcement has been observed. The reaction seems to depend on the power density of the beam. This correlates with Devletian’s experience of laser processing \textsuperscript{49} with a high power density beam in a keyhole mode.

2.3.3 RESISTANCE WELDING

Most of the information on resistance welding concerns Boron-Aluminum composites \textsuperscript{50, 51, 52, 53}. Though there is a claim of 90% of theoretical strength, for resistance spot welding of Boron-Aluminum composites, details of the basis of the claim are not clear \textsuperscript{50}. Also, the joint efficiencies for resistance seam welding range from 39.2 to 45.8 %. Failures in lap shear specimens, tested in the fiber direction, occurred at the edge of the spot by the filament breakage. In the transverse direction, failure occurred at the edge of the spot \textsuperscript{50}. Metzger \textsuperscript{64} observed that the average joint efficiency in Al-B spot welded to Al-B or aluminum alloys ranged from 14 to 38 %. In addition, the spot welded joints are sensitive to bending stresses, based on a low ratio of cross-tension to tensile shear strength \textsuperscript{64}. The average efficiencies, with a large overlap and very closely spaced spots, are in the range of 40 to 45% for Al-B composites \textsuperscript{64}. It is very difficult to portray resistance welding as a flexible process for joining composite materials, in
general, based on the above information, though it may be an acceptable process for some particular applications.

2.3.4 **SOLDERING AND BRAZING**

Though the brazing process could be attractive for joining composite materials, the formation of intermetallics, voids, precipitates, debonds and disordered fiber orientation near the interface can not be ruled out. Moreover the strength values reported, except in a few cases, are not satisfactory.

Most of the brazing work on composite materials was carried out on the Al/B system. The joint efficiencies, based on the tensile strength, ranged from about 49 to 88% \(^{64}\). It may not be appropriate to base the success of a brazed joint on tensile strength in view of the apparent increase in strength from the restraint effect. The brazed joints typically have a small thickness of the braze foil at the interface between the parent materials that are joined. When the joint is tested in tension, the small thickness of the brazed foil restrains deformation in the base metal, thereby promoting a higher tensile strength. Thus, the use of shear strength and bend strength will be more reliable. In most brazing applications, the filler metals used for Al/B composites were Al-8Si or Al-12Si \(^{64}\). The brazing temperatures and times were typically 580 to 590 °C for 5 to 30 minutes.

50 % Boron/6061 Al composites were brazed with aluminum-silicon (Alcoa 713 and 718) brazing foils \(^{54}\). The shear strength of the joint was calculated to vary between 11.75 to 15.7 ksi. In tension, failure occurred in the composite at the edge of the joint.
The base material had a tensile strength of 160 ksi. Results of lap joints have shown a tensile strength of 74 ksi, which is about 46% of the base metal strength. A butt joint design with doublers provided a strength of 119 ksi. With improvements in the joint design involving reinforcing straps to reduce stress concentration, a joint efficiency of 90% was obtained. Silicon precipitates at grain boundaries in the microstructure appear to lower the ductility. Bend tests indicated shear strength values of 11.91 to 13.77 ksi.

The brazed joints had an endurance limit 40% of that of the Al/B composite material. Metzger observed that the brazing cycle of 5 to 15 min at 590°C resulted in a loss in tensile strength (possibly due to thermal degradation of properties) of 10 to 20% for Al/B composites.

It is reported that the presence of magnesium in the brazing foil facilitates the wettability of the base metal in the Al-Graphite composite/6061 Al alloy system. Increases in time and temperature caused void formation at the interface. In the brazing of another composite material, 6061 Al alloy reinforced with 10 Vol% alumina short fibers, two types of brazing foils were used: i) a 4045 (Al-10%Si) and ii) a clad foil with 3003 (Al-1 to 1.5% Mn) as the core and 4045 as the cladding. The tensile strength of the composite to 6061 Al joint, brazed with the 4045 foil, decreased from 130 Mpa (18.65 ksi) to 60 Mpa (8.6 ksi) as the brazing temperature was increased from 580°C to 610°C. Silicon and magnesium enrichment at the fibers was noted. Typical problems in this system were, porosity, disordered fiber arrangement, unbonded regions and "grain boundary erosion". Brazing with the clad foil, however, showed a peak strength of 200 Mpa (28.69 ksi) at a brazing temperature of 600°C. The higher strength was attributed to a smaller volume of the liquid phase at the brazing temperature.

The composite (5% alumina fiber) to composite joint brazed with the clad foil had tensile
strengths ranging from 230 to 282 Mpa (33 to 40.65 ksi). In another study vacuum brazing was used to join 20 Vol% SiCw/6061 Al composite sheets, in the lap joint configuration, with Zn-Al and Al-Si (718 eutectic) foils at 600°C and 10^-5 torr vacuum for 5 minutes. Some significant results of this study include a lower hardness in the brazement than in the base metal, minimal penetration of the braze alloy into the base metal and failures in the base metal near the edge of the overlap. The strength (probably lap shear strength) values reported varied from 3.2 to 7 ksi.

In the soldering process, a Zn-5Al-5Cu (380°C melting temperature) filler material provided poor strength at a level of 10% of the brazed joints for an Al/B composite.

2.3.5 CAPACITOR DISCHARGE WELDING

In the capacitor discharge welding process, an arc, powered by a capacitor bank, is initiated between two parts, one of which is stationary and the other moving under a force towards the former. This arc heats both faying surfaces to the melting temperature. As the parts meet, solidification occurs and excess molten metal is expelled. This process is a rapid solidification process. In the joining 20 to 40% SiCp/6061 Al to itself and to 6061 Al, Devletian found that metallography revealed sound welds with no formation of Al4C3. Tensile testing results were not reported. The failure location in tension was outside of the fusion zone. No further comments can be made on this process due to lack of mechanical property data, however, this process may have good potential for joining composite materials although with somewhat limited flexibility.
2.3.6 **INERTIA WELDING**

In the inertia welding process, one of the two pieces to be joined is held in a stationary position, while the second piece, attached to a flywheel, is rotated at a high speed. The driving power is removed at a pre-determined speed and the pieces are forced together under axial pressure. It is a solid state joining process and is suitable for the joining of engineered materials such as composites. In a study on the joining of 20 Vol% SiCw/6061 Al tubes to themselves and to the matrix material, nearly defect free welds with excellent bonding were reported. Hardness traverses across the inertia welded joint region indicated nearly uniform hardness unlike the steep variation reported near the heat-affected zone of a fusion welded joint region. The absence of Al₄C₃ was confirmed by x-ray diffraction. The tensile strength reported for the composite material, in the post weld heat treated condition, was 72 ksi which is about 86% of the parent material strength. The composite to 6061 Al joint, in the post weld heat treated condition, was reported to have a tensile strength of 46.9 ksi. However, there was no information on the failure location and the composite joint showed a 2% elongation compared to the 3.2% elongation for the parent material. Inertia welding requires excessive deformation at the interface and may not be applied to components where minimal deformation is required. It also needs post-bond machining, due to the deformation.

2.3.7 **DIFFUSION BONDING**

Though diffusion bonding is one of the prominent processes used in the primary fabrication of composite materials, it has been used to a very small extent for secondary
fabrication. It is a joining process wherein coalescence between two clean and closely fitting parts is obtained under the application of heat and pressure. There are several advantages of the diffusion bonding process for the joining of composite materials. These include:

i) processing temperature lower than that in fusion welding and as a consequence;

ii) unwanted reinforcement - matrix reactions are eliminated;

iii) strength degradation of the reinforcement is reduced;

iv) an excellent control of the reinforcement position can be maintained.

There are two variants in the diffusion bonding process i.e., deformation bonding and diffusion bonding, with the latter itself being divided into solid state and liquid phase diffusion bonding processes. Deformation bonding relies on extensive plastic flow to disrupt oxide films and achieve metallic bonding. In the solid state diffusion bonding process, all the reactions take place in the solid state, while in the liquid phase diffusion bonding process, a liquid phase generated either through a low melting eutectic interlayer or due to the eutectic reaction between two components of a eutectic system helps the bonding process.

2.3.7.1 DEFORMATION BONDING

The important factors for deformation bonding can be summarized as:

i) degree of deformation;
ii) surface condition ;
iii) temperature ;
iv) composition ;
v) prior cold work ;
vi) crystallographic orientation ;

In the case of aluminum alloys, deformation is very important for solid state welding. Vaidyanath et. al. demonstrated that for roll bonding aluminum at room temperature virtually no bond strength could be obtained for deformations less than 40%. Figure 2 (Ref. 68) shows that this threshold deformation is a function of temperature and can be lowered as the temperature is raised. However, attainment of threshold deformation itself may not be viewed as sufficient to achieve full base material strength. McEwan and Milner 69 state that though the threshold deformation for welding (roll bonding) of aluminum at room temperature is 40%, the strength of solid metal is attained only at a deformation of 70% i.e., the strength of the weld increases with deformation. Hauser 70 et. al, suggest that the cleaning procedure, apart from temperature, also effects the threshold deformation. Surface condition effects deformation due to the need for deformation assisted metallic contact. The basic purpose of the threshold deformation is to obtain intimate contact at the contacting surfaces and disrupt the oxide film. The variation of elastic limit with temperature dictates the amount of deformation necessary to obtain permanent surface matching. In addition, elastic recovery can break the welds on removal of the welding pressure.
Fig. 2 Variation of threshold deformation with temperature for aluminum (Ref. 68).
2.3.7.2 SOLID-STATE DIFFUSION BONDING

Solid-state diffusion bonding does not involve melting but may include "limited macroscopic deformation of parts" during welding. This process occurs in the following steps:

i) contamination removal and surface cleaning;
ii) dissolution of oxide;
iii) yielding and creep of surface asperities;
iv) elimination of voids by vacancy diffusion;
v) formation of atomic bonds across the interface.

A three-stage model describes this process as involving yield and creep deformation of asperities resulting in a joint of grain boundary with voids, diffusional elimination of voids, grain boundary migration and pore elimination by volume diffusion.

Diffusion bonding conceptually is not accompanied by significant deformation.

The important factors for diffusion bonding can be stated as:

i) welding pressure;
ii) surface condition;
iii) temperature;
iv) time;
v) intermediate materials;
vi) reheat treatment.
As has been already stated, surface condition is very important for diffusion bonding in view of the need to bring atoms on the matching surfaces close to their interatomic distance. Figure 3 (Ref. 71) shows the typical surface roughness and contaminants present on a metallic surface.

Considering that an average oxide film on aluminum alloys varies from tens to thousands of angstroms and the surface finish achieved in finish turning can be 1 to 3 microns, one can visualize that actual contact of the asperities will be very small compared to what it appears to be 72. Since solid-state diffusion bonding relies on interatomic forces once the atoms are brought to distances where interatomic forces come into play, the need for very good surface finish and yield and creep of asperities becomes very clear. As Figure 3 shows, metallic surfaces are usually covered with soil, grease, dirt and other contaminants, oxides and other reactional products and are also rough 71. In the case of aluminum, the adsorbed contaminant is very important and must be removed to improve bonding 69. The most critical adsorbed component for aluminum alloys is water vapor. Nicholas and Milner 74 reported that when commercial purity aluminum and super purity aluminum were self-deformation bonded, the former bonded more easily than the latter. However when baked out (500 °C) surfaces were used, they did not see any appreciable difference. A process of machining in vacuum and baking out at 500 °C resulted in a reduction of threshold deformation at room temperature for super purity aluminum from 25 to 10%. They 68, 74 assumed that a certain amount of water vapor is trapped at the interface. With a water layer several molecular layers thick, one of two things can happen:
Fig. 3 Typical surface roughness and contaminants on a metallic surface. (Ref. 71).
i) it does not react with aluminum and in such a case welding is not possible due to excess water molecules;

ii) the hydrogen atoms are liberated, form molecules and prevent bonding.

Cline 67 observed that "even heavily oxidized surfaces could be bonded if they were first baked out and allowed to cool in a desiccator to prevent reabsorption of contaminants". Thus, for metallic bonding to take place, these contaminants must be removed. Contaminant and oxide removal by chemical and/or mechanical means achieves the first step. Temperature helps in the desorption of contaminants and in obtaining intimate contact through yield and creep deformation 65.

An increase in temperature will enhance atomic diffusion coefficients, decrease the material yield strength and promote desorption of contaminants from the surface 67. The diffusion coefficient is related to the temperature in the following way 65:

\[ D = D_0 \exp \left( -\frac{Q}{RT} \right) \]  

where:

- \( D \) = diffusion coefficient,
- \( D_0 \) = constant,
- \( Q \) = activation energy for diffusion,
- \( R \) = gas constant,
- \( T \) = temperature.

The above equation indicates that the diffusion coefficient increases exponentially with temperature. Due to the temperature dependence of material yield strength, yielding and creep deformation of asperities will be easier and can be achieved at lesser deformation.
with an increase in temperature. Desorption of contaminants is improved at higher temperatures \(^6^7\) as the kinetics involved are a temperature dependent phenomenon.

Time is considered in the diffusion bonding calculations through the solutal diffusion distance given as \(^6^5\):

\[
X = C (D t)^{1/2} \tag{3}
\]

where:

- \(X\) = diffusion distance,
- \(C\) = a constant,
- \(t\) = time.

The selection of time is dictated by the process and system considerations. Equation 3 gives the time necessary to completely diffuse a diffusion aid at the interface in the form of an interlayer, vapor, electrodeposited or ion-deposited film. This is based on the diffusion coefficient which depends on the system (the type solute/solvent employed) and the bonding temperature. In addition, formation of intermetallic compounds at the interface have to be taken into account. The thickness of the intermetallic compound, which is important from strength considerations, depends on time and system considerations. In spite of the above, time has a limited role to play in diffusion bonding from economic considerations.

It is reported that alloying elements which are likely to increase the base metal hardness will also increase the threshold deformation \(^6^7\). Base metal composition directly affects the diffusion bonding process through factors such as oxygen solubility and the standard free energy of formation of oxide. Brayant \(^7^5\) divided metals into three
categories based on the above information. Metals with a high oxygen solubility and a high (positive) free energy of formation of oxide were considered to be successfully hot isostatic pressure (HIP - a diffusion bonding method\textsuperscript{76}) welded, in spite of the presence of oxide film. Metals with a very low oxygen solubility and a very low free energy of formation of oxide were considered to exhibit greatest difficulty for HIP welding due to the presence of oxide. Metals with intermediate oxygen solubility and an intermediate free energy of formation of the oxide were placed into an intermediate category. Aluminum has a very low oxygen solubility (less than 0.01 atom percent) and a very low free energy of formation of the oxide (less than -350 Kcal/mole) and thus is considered difficult to diffusion bond\textsuperscript{75}. In addition, in diffusion bonding, the composition of the system also dictates the formation of voids at the interface. If the system consists of components whose diffusivities differ widely at the bonding temperature, the formation of voids at the interface has to be taken into account\textsuperscript{77,78}.

There are several reports of using intermediate materials as diffusion aids in the diffusion bonding of aluminum alloys to themselves and to other materials\textsuperscript{67,70,72,73,79,81-84,86,87,90}. Typically interlayers are used for a number of reasons such as:

i) improving plastic flow;

ii) attainment of intimate surface contact\textsuperscript{70};

iii) increasing diffusion coefficients;

iv) provision of easily removable oxides;

v) minimizing or eliminating intermetallics;

vi) in the joining of dissimilar materials;
vii) interlayers make provision of liquid phase through eutectic melting and a reduction of bonding temperature 67, 80.

As opposed to brazing, where the joint strength and working temperature may be restricted by the formation of intermetallics and the lower melting temperature of the braze foil, the joint strength and the working temperature of the component in diffusion bonding may not be restricted by the interlayer 85. Iwamoto et. al. 87 reported that the joint strengths were poor if mild steel and aluminum were diffusion bonded without an interlayer. Diffusion aids can be in the form of electrodeposited, plasma sprayed, vacuum deposited, vapor-deposited, ion-deposited coatings or loose foils. The form in which the diffusion aid is applied depends on the system under consideration. Each form of application has certain advantages and disadvantages. The typical problems that have to be overcome while utilizing interlayer materials include poor adhesion, peel off, Kirkendall porosity due to large differences in diffusivities between the base metal and the interlayer material, difficulties in electrodeposition and the formation of brittle intermetallics. Electrodeposition has the advantages of providing clean and oxide free surfaces with the right choice of diffusion aid for the system under consideration. Thus, this will be of great use in aluminum base alloys where a tenacious oxide film interferes with the bonding. Moreover, the deposition can be made adequately strong to initiate failure either in the substrate or in the deposit rather than at the interface 79. Hot hollow cathode vapor deposition appears to achieve better bond strengths than electrodeposited interlayer material in the case of aluminum alloys. It produces highly adherent films without intermetallics on aluminum alloys 86. With a loose interlayer foil shear deformation can occur through the thickness helping intimate contact between matching surfaces 70, 88. Barta 73 observed that loose interlayer foil materials may be better for
high temperature diffusion bonding processes than for low temperature processes. With a softer interlayer, adequate deformation of the interlayer can be achieved with a minimal load, that can provide good surface matching at the parent material interfaces. Also the presence of a thin interlayer can improve the joint tensile strength due to the restraint effect as explained in section 2.3.4.

The thickness of the interlayer to be used depends on the chemistry, kinetics and other factors such as formation of voids, intermetallics, their thickness and properties (ductile or brittle) of the parent material - interlayer system under consideration. Some examples of the above include a higher void concentration at 6061 Al interfaces than that for 1100 Al and formation of ductile intermetallics in the silver-aluminum system compared to brittle intermetallics in the copper-aluminum system. McEwan and Milner studied the ductility of the intermetallics in the silver-aluminum and copper-aluminum systems. They observed that in the former case the intermetallic was ductile and in the latter case the ductility of intermetallic depended on the thickness with thin and thick intermetallics being ductile and brittle respectively. They also showed that in the case of aluminum-copper and aluminum-silver multilayered specimen, the joint strength decreased with the intermetallic thickness for the former case whereas for the latter it did not change with the intermetallic thickness. However Naimon et. al. reporting on the diffusion bonding of aluminum to stainless steel with silver as a diffusion aid observed that " an intermetallic width of approximately 4 µm severely embrittles the joint ".

They also indicated that the joint strength decreased linearly with the intermetallic thickness (between 1 to 21 µm) and that the impact strength dropped abruptly with increasing intermetallic thickness (between 1 to 9 µm). Thus the formation and thickening of intermetallics is important from diffusion bonding consideration.
The thickness of the diffusion aid also depends on basic process considerations such as the time and temperature of bonding. For a particular system under consideration the thickness of the diffusion aid should be optimum. Too large a thickness would demand an unnecessarily high temperature or time of bonding and contributes to a large intermetallic thickness. Too small a thickness may make surface matching difficult and may have cleanliness problems. The type of interlayers used in the diffusion welding of aluminum alloys include silver 67, 73, 79, 82, 84, 86, 87, 90, copper and copper alloys 70, 73, 79, 82, nickel and nickel alloys 73, 79, 87, zinc and alloys 73, 79, 90. Tylecote, reviewing Crane et al.'s joining of aluminum to stainless steel, observes that silver plating is superior for the above application in view of the following reasons:

i) the bonding temperature is close to the silver oxide dissolution temperature (in air), which makes it's dissociation fairly easy;

ii) it is very unlikely that brittle intermetallic compounds will be formed in the silver-aluminum-and other constituents of steel system.

Post heat treatment can be used to restore the mechanical properties of the base material and the joint. In the case of precipitation hardenable aluminum alloys, the bonding thermal cycle may cause precipitate re-solution or coarsening based on the thermal cycle employed. In such cases, it may be possible to restore the base material mechanical properties by post-bond heat treatment. Cline 67 reported strength improvements after post bond heat treatment in deformation and eutectic diffusion bonded 6061 Al alloys. Barta 73 reported a 50% strength increase after post heat treatment (T6) for a diffusion bond between 7075 Al base materials with a 7072 Al clad.
Thus, several interesting examples can be found in the literature regarding diffusion bonding of aluminum alloys. In spite of that, diffusion bonding has not been utilized to a great extent in the secondary fabrication of aluminum based metal matrix composites. This may be understood in terms of Metzger's observation, that, to achieve a 100% joint efficiency in the diffusion welding of aluminum alloys, a good amount of plastic deformation is required, even at very high temperatures (close to the solidus). He maintains that it is not possible to obtain a high strength diffusion bonded joint with fully compacted 40-50% B fiber reinforced Al composite materials in view of anticipated fiber damage due to the matrix plastic deformation required. However, Olster and Jones demonstrated in the case of stainless steel wire reinforced 2024 Al composites, that, it is possible to obtain good joint efficiency (average 87%) with diffusion welding. They used a bonding temperature of 538 °C, pressures of 84, 101, and 123 Mpa (12.17, 14.64 and 17.82 ksi) and times ranging from 1 to 1.5 hours. Salkind carried out diffusion bonding of Al3Ni whisker-reinforced Al matrix composite at temperatures ranging from 500 to 525 °C, pressures from 8 to 20 ksi and times from 1/2 to 1 hour, with lap and scarf joint configurations. They reported tensile strengths from 19.2 to 43.4 ksi and a failure in the base metal in a large number of cases. Hersh performed resistance diffusion bonding of B/Al composites to titanium. He reported a 50% (of base metal) static lap shear strength in the temperature range of 70 to 250 °F and 40% at 600 °F. He further reported that a fatigue limit of 50% of room temperature strength could be attained for room temperature applications. Enjo et al. self-diffusion bonded 15 Vol% alumina short fiber reinforced 6063 Al composite. They studied the effects of varying surface finish of the faying surface and interlayer materials on the joint properties. They obtained the best tensile strength with an electropolished surface finish. Wire brushed and turned surfaces yielded still lower bond strengths. The
interlayer materials used were silver, copper and an Al-Cu-Mg alloy. The bonding temperatures ranged from 1022 to 1130 F (550 to 610 °C), pressure from 145 to 290 psi (1 to 2 Mpa) and a time of 30 minutes. They reported bond strengths ranging from 14.2 to 27.24 ksi (98 to 188 Mpa). In most cases the fracture occurred at the bond interface. They observed that the bond strength and deformation were improved with insert materials. The temperatures and times point out, that, in this case the bond was associated with the presence of a liquid phase.

The above reports suggest that in the case of aluminum alloys solid-state bonding may require insert materials, an appropriate surface finish, useful techniques to deal with the oxide layer and most likely the need for extensive plastic deformation in order to provide good joint strength. The techniques to deal with oxide layer may involve laborious procedures such as electro/plasma/ion/vapor deposition of films. The joint design should be adequate for the plastic deformation required. This also necessitates post bond machining. In the case of aluminum based composites, the effects of extensive plastic deformation on the reinforcement-matrix interface and reinforcement morphology is not clear. Thus, it would be better if a process, which can bond aluminum based composites with a low deformation and which does not require extensive oxide removal procedures, is available. Liquid-phase diffusion bonding provides such opportunities.

2.3.7.3 LIQUID-PHASE DIFFUSION BONDING

Duvall et al. observe that the liquid-phase diffusion bonding can produce high strength diffusion bonds in heat resistant materials without applying substantial pressure during bonding. Liquid phase diffusion bonding has also been applied to join aluminum
based alloys and metal matrix composites. The process relies on the formation of a liquid phase, either through eutectic melting of a system of two eutectic forming components at or slightly above the eutectic temperature or the melting of a low melting eutectic interlayer sandwiched between the components to be joined. As the parts are held at the bonding temperature, composition changes due to rapid interdiffusion result in isothermal solidification. The solidified bond region consists of a solid solution nearly similar in composition to the base metal. The bond can be homogenized by additional time at temperature. Thus the formation of brittle phases in the bond region as in brazing or other joining techniques is restricted. Transient liquid phase (TLP) diffusion bonding, thus, combines the advantages of solid state diffusion bonding with the manufacturing ease of conventional furnace brazing. In addition, the bonding operation is relatively simple. Also, several reports in the literature point out the fact that the liquid phase formed through interdiffusion through the oxide film helps in the removal of the oxide film ever present on aluminum alloys. For the reasons discussed above, TLP bonding, has been chosen for joining SiC/Al MMC's in this study.

2.3.8 TRANSIENT LIQUID PHASE DIFFUSION BONDING

TLP process has two variants. In the first process, a eutectic system (comprised of the base metal to be joined and the diffusion aid) is held isothermally under slight compression at a temperature slightly higher than the eutectic temperature. The diffusion aid can be a pure metal or an alloy system capable of forming a eutectic system with the base material at the bonding temperature. The diffusion aid can be an interlayer foil or an electodeposited/plasma deposited/ion deposited/vapor deposited coating.
Equilibrium thermodynamics dictates that under such conditions a eutectic liquid is formed. The liquid phase thus formed attains an equilibrium composition eroding the base material in the process. It spreads and wets the joint under appropriate conditions. Assuming that microscopic equilibrium prevails at the interface, with continued holding at this temperature the liquid solidifies due to diffusional loss of solute to the solid with which it is in contact. With continued depletion of solute and the consequent solidification of the solute-lean liquid, the solid-liquid interface moves into the liquid until the liquid isothermally solidifies. The last liquid to solidify will have attained a composition corresponding to the equilibrium maximum solid solubility at the temperature under consideration. At this stage solidification will be complete, and further continued holding at the temperature results in homogenization of chemistry.

In the second process a low melting eutectic interlayer is held in between the metals to be joined. Upon heating the assembly to the bonding temperature (which is higher than the interlayer melting temperature), the interlayer melts and fills the joint region. Assuming microscopic equilibrium at the solid liquid interface, the liquid erodes the solid at the interface until the interface chemistry reaches those values predicted by the equilibrium phase diagram. Whereupon, the liquid homogenizes to the new chemistry. The process hereafter proceeds exactly like that in the above case. Due to continued holding at the temperature and consequent diffusion of solute into the solid, the solute-lean liquid in contact with the solid solidifies and the solid-liquid interface moves into the liquid until isothermal solidification is completed.
The second process seems to be similar to the brazing process but has distinct differences. Duvall et. al. analyzed the similarities and differences between brazing and the above variant. They suggested that the similarities existed only in the initial stages of the process. The distinct differences include:

i) the time of bonding in the case of the TLP process depends on the quantity of liquid to be solidified, the diffusivity of the solute and the thermodynamics of the system (which in the case of brazing is only a few minutes normally dictated by wettability and fill factors);

ii) the final composition at the TLP bonded interface is expected to be a single phase solid solution (unlike that in brazing which consists of a dissimilar braze foil compared to the base material);

iii) the non-equilibrium solidified braze structure may contain brittle intermetallics.

Figure 4 provides a schematic illustration of the TLP bonding process. Steps (a) to (f) illustrate the TLP bonding process with an interlayer of material B which forms a eutectic system with the base metal A to be joined. Steps (b) to (f) illustrate the TLP bonding process with a eutectic interlayer. In either case erosion of a certain amount of base metal is inevitable. TLP bonding is thus a diffusional process occurring in distinctive stages.
Fig. 4 Illustration of the TLP bonding process (Ref. 94).
Issac Tuah-Poku et al. modeled TLP bonding partly based on earlier work by Lesoult at the Carnegie Mellon University. They considered the processes occurring when the liquid phase is present and after the complete solidification of the liquid phase. The model did not consider the problems occurring in completely miscible systems. In completely miscible systems, the processes occurring in the solid state after isothermal solidification are important. This arises due to the difference in diffusivities of the different species. Large differences in diffusivities can give rise to Kirkendall porosity on the side of the faster diffusing species. Any model can only be considered complete if it can also take this into consideration and such a realistic model for TLP bonding has not been developed to date. In the discussion ahead, the model described earlier for TLP bonding will be presented and will be augmented by the necessary information obtained by other workers regarding theoretical studies of the formation and elimination of voids in solid state welds. Such a combination is likely to make the modeling of TLP bonding complete.

The assumptions made for the TLP bonding model include:

i) microscopic equilibrium at the solid-liquid interface;

ii) negligible convection in the liquid;

iii) $D_S$, $D_L$, the interdiffusion coefficients in solid and liquid phases to be independent of composition;

iv) the partial molar volumes of A and B in all phases are assumed equal (Fig 4).
Assuming a moving boundary problem involving solid and liquid phases, the process has been analyzed in four stages:

Stage 1. interlayer dissolution;
Stage 2. liquid homogenization;
Stage 3. isothermal solidification;
Stage 4. homogenization of bond region.

A fifth stage may be added to the above to include the elimination of voids for completely miscible systems with species with wide differences in diffusivities. The five stages presented are for the TLP process involving pure metals reacting to form eutectic liquid. In the case of the utilization of a eutectic interlayer for the TLP process, the melting of the interlayer should replace Stage 1. Stages 1 and 2 are controlled by diffusion in the liquid $D_L$ and steps 3, 4 and 5 are controlled by diffusion in the solid $D_S$. In addition, Stage 5 is controlled by the plastic deformation processes occurring at the temperature.

Diffusion conditions in stage 1 can be represented by Fick's law as follows:

$$\frac{\partial C_L}{\partial t} = D_L \frac{\partial^2 C_L}{\partial t^2} \quad (3)$$

where:

- $C_L$ = composition of B in the liquid,
- $D_L$ = diffusion coefficient in the liquid.

The solution is represented by:

$$C_L = E + F \text{erf} \left( \frac{Y}{(4D_Lt)^{1/2}} \right) \quad (4)$$
where: \( Y = \) distance across the bond region and \\
\( E, F = \) constants determined by the boundary conditions.

The driving force for continuous dissolution of B will be proportional to the difference \( C_{\beta} - C_{\alpha} \), which represents the concentration differential in the liquid phase, as represented by the equilibrium phase diagram at the bonding temperature. The total time required for the dissolution of the interlayer may be very short, since it is controlled by diffusion in the liquid, which is orders of magnitude faster than the diffusion in the solid state. The interface displacement can be represented by \( Y_1 \):

\[
Y_1 = K_1 (4D)_{\text{l}}^{1/2} \quad (5)
\]

where \( K_1 = \) alloy and temperature dependent constant \\
and \( Y_1 = \) interface displacement during stage 1.

Stage 2 is complex and has no known solution. This represents an inhomogeneous and supersaturated liquid with an average composition greater than the concentration of liquid in microscopic equilibrium with the solid at the bonding temperature. Thus, the primary phase at the solid-liquid interface is dissolved, in order to maintain microscopic equilibrium whenever the liquid composition at the interface exceeds the equilibrium concentration of the liquid. This causes dilution of the liquid since diffusion in the liquid is extremely fast compared to diffusion in the solid. Thus, the remaining liquid continuously homogenizes and the liquid zone widens. This process continues to erode the base metal until the entire liquid homogenizes to the equilibrium...
concentration, as dictated by microscopic equilibrium at the interface. The effective
diffusion coefficient at this stage may be a geometric mean between $D_L$ and $D_S$.

During stage 3, isothermal solidification occurs. The solid-liquid interface
changes direction and moves back into the liquid until the liquid completely solidifies.
The interface movement rate depends on the loss of solute from the liquid to the solid,
which is controlled by the diffusion of the solute in the solid $D_S$. The boundary
conditions in stage 3, can be represented as follows:

$$
C_\alpha = C_0, \quad Y < 0 \\
C_\alpha = C_{\alpha L}, \quad Y = 0 \\
C_L = C_{L_\alpha}, \quad Y > 0
$$

where:
- $C_0$ = initial composition of the base metal A,
- $C_\alpha$ = concentration of B in the solid,
- $C_{\alpha L}$ = composition of solid in equilibrium with
  the liquid at the bonding temperature as
given by the equilibrium phase diagram,
- $C_{L_\alpha}$ = composition of liquid at the bonding
temperature as given by the equilibrium
  phase diagram.

Applying Fick's law for diffusion of solute in the solid, in the case of one dimension:

$$
\frac{\partial C_\alpha}{\partial t} = D_S \frac{\partial^2 C_\alpha}{\partial x^2}
$$
where \( C_\alpha \) denotes the concentration of \( B \) component in the solid. The solution for the above equation which satisfies the boundary conditions in (6) can be given as\(^{94, 95}\):

\[
C_\alpha = M + N \, \text{erf} \left( \frac{Y}{(4D_s t)^{1/2}} \right) \quad (8)
\]

where \( M \) and \( N \) are constants determined by the specific boundary conditions. The interface displacement rate, from the above, will obey:

\[
Y_3 = K_3 \, (4D_s t)^{1/2} \quad (9)
\]

where \( D_s \) is the diffusion coefficient in the solid, \( Y_3 \) is the interface displacement during stage 3 and \( K_3 \) is a constant, which is a solution of the general equation \(^{94, 95}\):

\[
K_3 \exp \left(-K_3^2\right) \left\{ 1 + \text{erf} \, K_3 \right\} \sqrt{\pi} = \frac{C_{\text{sol}} - C_0}{C_{\text{La}} - C_{\text{sol}}} \quad (10)
\]

Since the right hand side of the above equation can be obtained with phase diagram data, \( K_3 \) can be obtained.

Stage four represents additional time at temperature to homogenize the solid and is controlled by the diffusion of solute in the solid. This stage occurs immediately after the isothermal solidification and is represented by an initial interface composition as given by the maximum solid solubility of the solute in the solid at the bonding temperature as given by the equilibrium phase diagram. There will be no presence of the liquid phase in this stage. Hereafter, additional time at temperature will cause the solute to diffuse down the concentration gradient in the base metal with nominal composition \( C_0 \). This being a
single phase region diffusion continues as long as the sample is held at a reasonable
temperature until ultimately the solute hump in the solid homogenizes completely.
Theoretically, this time depends on the holding temperature since diffusion in the solid is
a slow process.

The Stage 5 process can be modeled based on a similar situation in solid state
diffusion bonding. In order to model the elimination of voids after Stage 4, one has to
consider the following processes:

i) plastic deformation acts to reduce the size of the voids remaining after
stage 4;

ii) vacancy diffusion from closed microvoids to defects or free surfaces
under the thermodynamic driving forces of microvoid surface energy and
external pressure also reduce void size.

Garmong et al.97 assumed that the operation of both of these mechanisms
resulted in the elimination of voids. The microvoid collapse by plastic flow was modeled
as the shrinkage of a thick-walled hollow sphere under hydrostatic pressure. The inner
radius of the hollow sphere was the microvoid radius, while the outer radius was taken as
half the center-to-center separation between microvoids in the bond line.

The microvoid size was assumed to decrease by diffusion. Because of capillarity
and pressure, the chemical potential of a vacancy within the microvoid was considered to
be higher than that at sinks, grain boundaries or at free surfaces; so vacancies diffuse
away from the microvoid (that is the atoms will diffuse into the microvoid).
Each of these two processes has been expressed as the rate of change of radius with time. With the above information the time required at a given temperature and pressure to cause microvoid closure was estimated. Calvo et al. in a study on diffusion bonding of copper and aluminum, suggested that shrinkage and elimination of voids can be expected from matter flow from external sources, or a counterflow of vacancies from voids to the metal matrix. McEwan and Milner suggested that diffusional porosity can also be a severe problem for systems possessing a high degree of miscibility. Clay and Greenwood observed that hydrostatic pressure caused the closure of voids created during interdiffusion in a Cu-Ni system. The hydrostatic pressure was not considered to affect atomistic diffusional processes.

2.3.8.2 TIME FOR ISOTHERMAL SOLIDIFICATION

The maximum width of interlayer after dissolution and the erosion of the base metal, in the process of preserving microscopic equilibrium at the interface was given as

\[
W_{\text{max}} = W_0 \left( 1 + \frac{C_L - C_{\text{sat}}}{C_{\text{sat}}} \frac{\rho_B}{\rho_A} \right)
\]

where

- \( W_{\text{max}} \) = final maximum width of molten zone,
- \( W_0 \) = initial width,
- \( \rho_A \) and \( \rho_B \) = densities of A and B,
- \( C_{\text{sat}} \) = composition of liquid in equilibrium with solid of composition \( C_{\alpha L} \) at the interface and
- \( C_L \) = composition of the bulk liquid.
The time for isothermal solidification $t_3$, was estimated based on the following assumptions:

i) A pool of liquid of initial width $W_{max}$ is shrinking gradually over a time $t_3$.

ii) Mass balance at any given time during this stage, assuming

$$\rho_a = \rho_\beta \quad \text{is governed by}$$

$$WC_L = W_{max}C_L\alpha + 2\int DS \frac{dC}{dy} \quad (12)$$

where $W = \text{width at time } t$, $WC_L = \text{actual mass of interlayer}$, $W_{max}C_L\alpha = \text{initial mass of interlayer}$ and $\int \text{term} = \text{flow of atoms across one of the interfaces due to solute diffusion governed by } DS$.

The solution to the above equation is $77, 94$:

$$C(y,t) = C_\alpha L \left(1 + \text{erf} \left(\frac{y}{(4DS \ t)^{1/2}}\right)\right) \quad (13)$$

From the above the time $t_3$, was estimated to be $94$:

$$t_3 = W_{max}^2 \frac{C_L\alpha^2}{C_{\alpha L}} \frac{\pi}{16D_s} \quad (14)$$
The above equation gives the time for isothermal solidification. From equation (11), $W_{\text{max}}$ was estimated. Assuming $P = P_0$,

\[ \rho_a = \rho_s, \quad W_{\text{max}} = W_0 \frac{C_{\text{eq}}}{C_{\text{eq}}} \quad (15) \]

\[ t_3 = \frac{\pi}{16D_s} W_0^2 \frac{C_i^2}{C_{\text{eq}}} \quad (16) \]

Thus $t_3$ depends on:

i) $W_0$ = initial thickness of interlayer,
ii) $D_S$ = diffusion coefficient of solute in solid,
iii) $C_L$ = initial composition of molten filler metal and
iv) $C_0L$ = equilibrium composition of solid in contact with molten filler metal.

It is evident that time for isothermal solidification depends on diffusion coefficient of solute in solid and not in liquid.

2.3.8.3 TIME FOR ISOTHERMAL SOLIDIFICATION IN THE AL-SI-MG SYSTEM

4.96% Mg-12.95% Si-Al forms a ternary eutectic system at 555°C. At this temperature, the solidification sequence $^{108}$ is:

\[ L \quad \rightarrow \quad \text{Mg}_2\text{Si} + \text{Si} + \alpha \text{Al} \quad (17) \]
and the solid solubility of Mg and Si in Al are 0.85 and 1.1 wt% respectively.

The following assumptions will be made to simplify the ternary system to consider it as a binary Al-Si system for diffusion of silicon in the solid state:

i) The available Mg combines with Si to form Mg$_2$Si which gives an equivalency of 2.5% Mg$_2$Si - 10.5% Si-Al

ii) Si is present in monoatomic state to enable diffusion and not as (Si-Si) or (Al-Si) clusters.

Hence the system was treated as a binary system as: Al- 10.5 Wt% Si.

Taking $D_{Si \alpha Al} = 9.97 \times 10^{-9}$ cm$^2$/sec at 575°C, $C_0 = 0.6$ Wt% Si, $C_L = 10.5$ Wt% Si and $W_0 = 0.001" = 2.54 \times 10^{-3}$ cm,

t has been estimated from equation (16). This works out to 1.5 hours for the 0.001" interlayer. The time to isothermally solidify a 0.004" interlayer would be 24 hours.

2.3.8.4 PURE METAL INTERLAYER

A pure metal or an alloy interlayer, can be utilized if it conforms to the following requirements:

i) it should form a low melting eutectic with aluminum;
ii) it should have considerable solid solubility in aluminum at or slightly above the eutectic temperature;

iii) it should be ductile or easy to apply as a coating;

iv) the solute should have a reasonably fast diffusion coefficient in the solid aluminum;

v) it should not form an intermetallic at the bonding temperature.

Silver forms a low melting eutectic with aluminum (567 °C), has a very high solid solubility in aluminum at the eutectic temperature, diffuses faster in aluminum and is ductile. These factors strongly support the candidature of silver as an interlayer for liquid-phase diffusion bonding applications. However, silver forms intermetallics with aluminum. Evidence of extensive plastic deformation before fracture in silver-aluminum deformation bonds 69, with 20 micron (0.0008") intermetallic thickness, lends credence to the ductility of thin silver-aluminum intermetallic films. However, due to the fast diffusion and large solid solubility of silver in aluminum at the eutectic temperature, bond line porosity may result in TLP bonding. This may be minimized or eliminated through diffusion bonding or homogenization under pressure.

2.3.8.4 OXIDE LAYER

The tenacious oxide film always present on aluminum alloys interferes with the formation of metallic bonds across the interface in diffusion bonding of aluminum 100. Several mechanisms have been proposed to explain the removal of oxide film. Some of them are due to the shear displacements obtained between two micro-asperities in contact 98, chemical cleaning and an electroless or electrolytic nickel plate 101; (nickel reacts
exothermically with aluminum, and the formation of nickel aluminide initiates melting of a ternary eutectic resulting in sudden volume expansion of molten filler metal and violent fragmentation of oxide layer); reliance on the eutectic diffusion bonding technique which helps in the dispersing the oxide layer and promoting diffusion (the transient liquid phase breaks up the thin Al2O3 layer and promotes diffusion 67, 79), which may be occurring, probably, due to the solute diffusion through the oxide film in view of the chemical concentration gradient existing at the interface 70, 102, 103.

In addition, the role of magnesium vapor in promoting flow in an inert or vacuum atmosphere (thermodynamic calculations and and vacuum brazing experiments at temp > 1125°F (607 °C) confirm the above 104) is beneficial. Magnesium vapor can be detected at temperatures above 750°F (399 °C), as it vaporizes from Al-Mg-Si clad 105. Reports from the vacuum brazing of aluminum also indicate the effectiveness of magnesium vapor, generated at temperatures above 450 °C, in the removal of the oxide layer 106. When aluminum is heated, differential thermal expansion causes the oxide film to craze. If this occurs in vacuum, fresh aluminum is prevented from reoxidizing. During long term heating in vacuum, the aluminum oxide is sufficiently disrupted to permit welding of base metal by the molten filler metal 57.

Winterbottom et. al.105 suggested that for an Al-Mg-Si system with temperatures below 500 °C the concentration of magnesium at the interface is limited by dissociation of Mg2Si, while above this temperature the diffusion of magnesium in aluminum is the slow step causing depletion. However, there is a rapid increase in evolution of magnesium from the liquid phase, at the eutectic temperature, which wets the surface of the porous oxide. The surface is converted from that of oxide to that of clean Al-Mg-Si
alloy at 560 °C. This effect is interpreted as resulting from the flow of eutectic liquid through the oxide.

On the other hand, Eustathopoulos observed that "a physical effect may contribute to loss of compactness of the oxide layer arising from stresses at the metal-oxide interface which may occur owing to a relatively very large change in volume of specimen on melting, approximately 7%".

Enjo et al. confirmed the presence of oxide film at the interfaces of a diffusion bonded joint in aluminum. They observed in a wire brush cleaned surface a number of acicular (19.68 micro-inches (0.5 μm) in length) and granular inclusions (3.94 micro-inches (0.1 μm) in diameter), in a zone of 39.37 to 118.1 micro-inches (1 to 3 μm) thickness near the bond interface, and concluded them to be oxides. In the case of surface treatments incorporating electropolishing following wire brushing or grinding on a metallographic paper of 1200 grit, the number of oxide inclusions observed were quite few. They were partially broken up and were lying only along the bond interface. They also cited evidence of small grains at the bond interface in spite of the high bonding temperature and conclude that oxide pinning of grain boundaries may have resulted in those smaller grains. They indicated that, in the case of wire brushed and subsequently electropolished surface treatment, failure location in tension testing was located in the base metal, in spite of the presence of oxide inclusions at the interface. They concluded that roughening of interface helps disrupt the oxide film. Also there are a number of reports in the literature which indicate that good joint strength in diffusion bonded aluminum alloys could only be obtained with a wire brush finished bonding interface. Thus, one may
conclude that, though the oxide removal does occur in TLP bonding the mechanism by which it occurs is not clear.

2.3.8.5 LIQUID PHASE BONDING OF COMPOSITE MATERIALS:

In section 2.3.4, the work of Breinan and Kreider on the joining of Borsic/6061 Al composite materials was briefly presented. They utilized 0.001" and 0.002" Al-Si brazing foils at 580 and 590 °C, for times between 5 to 30 minutes. With the Al-Si eutectic brazing foil, this process would have caused erosion of base metal after the melting of the braze foil. Subsequently silicon diffusion would have caused isothermal solidification to occur. However, the times employed by them for the thicknesses used were not sufficient to cause complete isothermal solidification, as the subsequent sections in this report show. Niemann and Willie utilized electroplated copper to form ternary eutectic liquid with aluminum and silicon (the ternary eutectic temperature is 524 °C), in the casting alloy A356.0, and obtained average strengths (failure) to 34.5 ksi (238 Mpa), compared to a base metal tensile strength of 47.6 ksi (328 Mpa). They utilized a plating thickness of 100 to 200 micro-inches (2.54 to 5.08 microns), a bonding temperature of 980 to 1000 °F (527 to 538 °C) and a bonding time of one hour. Niemann and Garrett eutectic diffusion bonded Boron/Aluminum composites. In view of the possible damage due to the fiber-matrix interaction at the elevated temperature required for an Al-Si interlayer system, they utilized copper plating to obtain a liquid phase diffusion bond with an 1100 Al matrix. Aluminum and 33.2 wt% copper form a eutectic at 1018 °F (548 °C). Their thermal simulation tests indicated that they still had a strength degradation ranging from 16 to 19%, compared to 35% expected in a brazing cycle at 1100 °F (593 °C). They selected a bonding time of 7 to 15
minutes and a coating thickness of 787 micro-inches (20 μm), based on theoretical considerations. They reported that some amount of the eutectic liquid was squeezed out during bonding and the absence of CuAl₂ intermetallic, at the bond line. However, CuAl₂ precipitates can be seen in the microstructure. They also suggested that the copper distribution could be homogenized by a post-bond heat treatment. Tensile tests indicated a longitudinal tensile strength of 160 ksi (1103 Mpa) compared to an unexposed base metal strength of 185 ksi (1276 Mpa).

Enjo et al. diffusion bonded 6063 Al alloy reinforced with 15 vol% alumina short fibers. Their interlayer materials and the bonding temperatures suggest that the liquid phase diffusion bonding process was used. They used a 2017 Al-Cu-Mg alloy (0.003 " and 0.0012 " (75 and 30 μm) thick), silver (0.00024 " (6 μm) thick) and copper (0.0002 " (5 μm) thick) interlayers at temperatures of 1130/1112 F (610/600 °C), 1112 F (600 °C) and 1130/1112/1022 F (610/600/550 °C) respectively. These temperatures are above the melting or eutectic temperatures of the aluminum base material and the interlayer material systems at the bonding temperatures considered. They studied the effects of interlayer materials and surface finish at the bond interface, on the bond characteristics. At a bonding temperature of 1130 F (610 °C), surfaces electropolished with "10 vol % hyperchloric acid" (probably perchloric acid) at -4 °F (-20 °C) and 18V for 300 seconds gave the a joint strength of about 23.2 ksi (160 Mpa), which was superior to the strength obtained when either a wire brushed or as turned surface was used. They observed that the last two treatments caused a change in fiber direction at the interface and also resulted in the presence of small fiber fragments at the interface. Even in the case of electropolished surface finish, the bond strength decreased with further polishing time, probably due to an increase in the length of fibers.
protruding from the interface. In the case of joints bonded without an interlayer, the
tensile strength of the joint increased with the bonding temperature in the interval of 1107
to 1143 °F (597 °C to 617 °C). Also, the deformation at the interface increased with
the bonding temperature. The deformation, in this case ranged from 2 to 7%. When
insert materials were used, the bonding deformation decreased to 2%.

Thus, summarizing, TLP bonding offers attractive features like i) minimal
deformation, ii) elimination/disruption of oxide layer and iii) avoidance of unwanted
intermetallics or reactional products at the interface and minimization of degradation in the
reinforcement and at the reinforcement - matrix interface. However, the details of the
process have to be worked out for each system under consideration on its own merits.
CHAPTER III

3.0.0 OBJECTIVES

The main objective of this study was to study the feasibility of joining SiC reinforced aluminum metal - matrix composites by the transient liquid phase diffusion bonding process. The detailed objectives were as follows:

i) Conduct a thermal and thermo-mechanical simulation of the SiC_p/6061Al metal matrix composite and to study the effects of time, temperature bonding pressure and bonding deformation on the base material and to observe if any large variations in mechanical properties as evidenced by hardness variations and any debonding of the reinforcement - matrix is present as evidenced in scanning - electron microscopy and thus estimate any degradation occurring due to the process thermo - mechanical cycle.

ii) Select an interlayer material and its thickness for the liquid phase bonding based on preliminary studies, and obtain process parameters based on
preliminary studies, thermo-mechanical simulation and refine them with the bond experience.

iii) Characterize the bonds with optical and scanning electron microscopy and understand the processes occurring in the bonding through observation of microstructural features and fracture features.

iv) Perform electron probe microanalysis to verify the concentration profiles of the interlayer material.

v) Conduct optical microscopic and electron probe microanalysis to conclude that the liquid phase exists in the bonding process.

vi) Identify the important parameters for the attainment of better mechanical properties.
CHAPTER IV

4.0.0 MATERIALS AND EXPERIMENTAL PROCEDURES

4.1.0 MATERIALS

4.2.0 COMPOSITE MATERIALS

The composite materials used in this study are listed as below:

i) 25 Vol% SiC_p/6061 Al metal matrix composite extruded bar stock of size 0.375 " diameter in various lengths;

ii) 25 Vol% SiC_p/6061 Al metal matrix composite extruded bar of size 0.55 " diameter x 67 " length and additional random lengths.

These materials have been procured from M/S Advanced Composite Materials Corporation, Greer, South Carolina.
The composite material used in this study was manufactured by a powder metallurgy solid state route as outlined in section 2.1.3. Pre-alloyed powder of the chemistry listed in Table 2 was mixed with 26.5 Vol % grade '3' SiC particulate. The resulting mixture was hot pressed into a 12.08" (307 mm) diameter x 12.85" (326 mm) long billet. This operation was carried out under vacuum and at a temperature between the liquidus and solidus temperature of the 6061 Al alloy. The temperatures and times are considered proprietary and are not available for publication. The vacuum hot pressed billet was machined to 12" (305 mm) diameter x 12.5" (318 mm) long. The billet was further extruded in two steps to 3" (76.2 mm) diameter x 7.5" (191 mm) long bar and then to 0.55" (14 mm) diameter. Most of the material used for this study utilized the 0.55" diameter bar stock. However, in some cases a further extruded bar stock of 0.37" (9.4 mm) diameter was also used.

Fig. 5, shows the optical microstructure of the 25 Vol% SiC particulate reinforced metal matrix composite material (0.37" diameter bar stock) used in this study (transverse cross-section, perpendicular to the extrusion direction), at a magnification of 400 X. The random distribution of the particulate material can be observed. Fig. 6 shows the microstructure, in the extrusion direction, of the 25 Vol% SiCp/6061 Al metal matrix composite (0.55" MMC). Fig. 6(a) shows the particulates in the extrusion direction at 4000 x and Fig. 6(b) shows an enlarged view of two particulates at 30000 x. Extrusion results in the fracture and preferential alignment of the particulate material. Extrusion also results in small bands of particulate free zones, which may be remnants of the base material powder particles elongated by extrusion.
Fig. 5 Optical microstructure of 25 Vol% SiCp/6061 Al MMC at low magnification (Note random distribution and sizes of the particulate material and lack of delineation of grain boundaries).
Fig. 6  Microstructure of a heat treated 25 Vol% SiCp/6061 Al MMC, (a) at 4000 x shows particulates in the extrusion direction and (b) at 30000 x shows a close view of two particulates.
Mohn et. al \(^1\), obtained the size distribution of particulates in an instrument grade SiC reinforced Al MMC and indicated that the particle sizes ranged up to 0.00035" (9 μm), with a mean size of 0.00014" (3.5 μm). As for the material used in this study, the manufacturer \(^{109}\) indicates that the SiC was grade '3' and probably had a size distribution mean around 0.00012" (3 μm).

Fig. 7 shows a scanning electron micrograph of a 20 Vol% SiCp/6061 Al MMC extruded material (received as 0.100" thick sheet; not used in this study) at a magnification of 8000X. The randomness of the particulate material distribution and sizes can be clearly seen. The alteration of the particulate morphology, as witnessed in the above figure, is really noteworthy. No debonding at the reinforcement - matrix interface can be observed. It is not possible to observe the grain structure in the Al matrix.

4.3.0 INTERLAYER MATERIALS

In this study two types of interlayer materials were used. An Al-Si-Mg ternary alloy made by Ribtec, Columbus. The composition of this interlayer as analyzed by CTL Engineering Inc, Columbus, is listed below:

- Aluminum : 75% (approximately),
- Magnesium : 4.46% and
- Silicon : 18.45%.

Fig. 8 shows the liquidus surface of the Al-Mg-Si system. A ternary eutectic at temperature of 559 °C and a composition of 5% Mg and 13% Si can be noted.
Fig. 7 20 Vol% SiCp/6061 Al MMC (received as 0.100" thick sheet) at a high magnification, Keller's Etch (Note particulate morphology and no noticeable debonding at the particulate-matrix interface).
Fig. 8  Al-Si-Mg ternary equilibrium phase diagram, liquidus surface (Ref. 110).
The Al-Si-Mg system was chosen to take advantage of the magnesium for oxide removal (see section 2.3.8.4). However this system introduced additional complications as described in a succeeding chapter, and was subsequently discontinued from full investigation.

A 99.9% silver interlayer foil of 0.001 " (25.4 microns) thickness obtained from Johnson Matthey, was used as a second interlayer. This interlayer was obtained to carry out liquid phase diffusion bonding based on the Ag-Al system. Fig.9 shows the Ag-Al eutectic at a temperature of 1053 F (567 °C) and a composition of 28 wt% Al. The Ag-Al system has some unique advantages. It can be treated as a binary system if we ignore the presence of a alloying elements in the Al matrix. A limited amount of diffusion data is available and in those cases where diffusion data is not available, some close approximations can be made based on simple assumptions. The interlayer material is ductile (can provide good surface matching at the interface) and easily available.

4.4.0 EXPERIMENTAL PROCEDURES

4.4.1 THE GLEE BLE 1500 SYSTEM

The Gleeble 1500 is a computer controlled thermo-mechanical system manufactured by Duffers Scientific, New York. Figure 10(a) provides an overview of the control cabinet and the computer that controls the machine. For a detailed description of the operating procedures of the machine, the thermo-mechanical cycle can be divided into two parts:
Fig. 9  Ag-Al equilibrium phase diagram (note the binary eutectic at 567 °C and 28 Wt% Al) (Ref. 110).
i) a thermal system, which includes the resistance heating and the temperature monitoring and controlling functions;

ii) a mechanical system which employs servo-controlled hydraulic functions for application and control of mechanical cycles, in conjunction with a load cell for measuring the forces employed.

For each specific job, the thermal and mechanical cycles required and their times of application are programmed into the control computer. The computer controls the specimen heating cycle. The specimen is heated by means of resistance heating with power supplied from an alternating current transformer. A total of nine heat settings built into the transformer give flexibility for varying sample sizes and materials. Since the equipment is used for studying highly conductive aluminum base alloys through relatively resistant stainless steels and nickel base alloys, this versatility in heat settings is very useful. Temperature measurement is accomplished through a pair of thermocouple wires percussion welded to the specimen. The type of thermocouple wires used depends on the application, i.e., the material system under consideration. For example, in the case of lower melting temperature materials like aluminum base alloys, chromel/ alumel thermocouple wires are used and for titanium base alloys Pt/Pt-13% Rh wires are used. The computer compares the specimen temperature with the set temperature and makes adjustments to the heating rate accordingly. The program can be plotted using a strip-chart recorder with a provision for recording three variables at various chart speeds. The machine is also equipped with a Datalyzer control to record and playback events occurring in a high speed testing environment. Unlike those in the indirectly heated systems like induction heating or furnace heating where uniform temperatures over the whole length of the specimen can be expected, in the resistance heated specimen, the length of the hot
zone i.e., the length over which the programmed temperature remains more or less constant or steady, depends on the type of material chosen to study. For example, in aluminum base alloys the hot zone is expected to be long whereas in stainless steels the hot zone may not extend beyond a small distance from the thermocouple location. A temperature gradient exists in the specimen from the thermocouple location to the point where it is supported in the jaws.

Fig. 10 (b) shows a view of the vacuum chamber without any specimen. The specimen is held (with additional fixturing) in a pair of water cooled copper jaws which are built into a moving and a fixed jaw. The moving jaw is connected to the hydraulic system through a load cell. The specimen chamber can be evacuated using a mechanical rough pump and a diffusion pump (for applications requiring a high vacuum) provided with a liquid nitrogen trap. The mechanical system can be operated with a stroke controlled mode or a force controlled mode both with the computer and manual controls. In the stroke controlled mode the hydraulic system controls the displacement of the moving jaw whereas in the force controlled mode the hydraulic system maintains the programmed force on the sample.

4.4.2 FIXTURE DESIGN

The specimen halves were held in a pair of split jaws which may be made of stainless steel. In a very few cases copper jaws with stainless steel inserts were also used. These jaws, holding the specimen, are clamped against the water cooled copper wedge jaws built into the machine. Fig. 11 shows the arrangement of specimen holding with a pair of split stainless steel hot jaws.
Fig. 10  (a) An overview of the Gleeble 1500 system showing the control cabinet and the control computer, (b) the moving and fixed jaws in the vacuum chamber.
Fig. 11  A view of the vacuum chamber showing the arrangement of the specimen in the stainless steel hot jaws.
These hot jaws are provided with M10 x 1.5 internal threads. The specimen to be used in the Gleeble, with the hot jaws, obviously also has mating M10 x 1.5 external threads. The threading in the hot jaws prevented the sliding of the specimen inside the jaws during the motion of the movable jaw. The threading in the hot jaws is located at the far end i.e., away from the bonding interface. This necessitates the use of specimen of about 1.5" length for each of the movable and fixed jaws, necessitating about 3" material for each bonded sample. As the composite material is very expensive, a second pair of stainless steel fixtures were designed and fabricated, with a two-fold purpose: 1) conservation of the expensive composite material and 2) serving as transition pieces for converting the requirement of having a metric thread in the specimen.

These fixtures were provided with an M10 metric external thread on one end and an 3/8 " 16TPI, internal inch thread on the specimen holding side. Fig.12 shows the arrangement for holding these shorter length specimen with this type of fixturing. With the arrangement shown in Fig.12, specimen halves of only about 1" length were used.

For diffusion bonding, the alignment of the specimen halves with the axis of the movable jaw was very important. Though the presence of two threads complicates the alignment in the second fixture, precision machining of the fixture, to some extent, solves the problem. The alignment of the specimen can also be complicated by the way in which the threads are cut. For example, hand die cut threads will have a lot of misalignment and require that the bonding interface diameter and squareness be attained with reference to the thread axis. It was always observed that the machining of the threads (with the machine) and the bonding interface in the same setting alleviated the misalignment to a very large extent.
Fig. 12 An arrangement of the specimen held in the stainless steel fixtures with the fixture themselves held in the stainless steel hot jaws.
4.4.3 SPECIMEN DESIGN

As the information provided in the above part illustrates, several factors enter the specimen design stage. The primary concern in the specimen design is the thermal load that has to be sustained by the Gleeble for the long diffusion bond cycles that are necessary for this experiment. Aluminum, being a high conductivity material, requires a large thermal load to heat to the elevated temperatures required in the liquid phase bonding. Though 25 Vol% silicon carbide decreases the electrical conductivity of the composite, the fact that it still contains 75 Vol% of an aluminum alloy has to be still borne in mind in the specimen design. The length of the specimen has been fixed on four considerations i.e., fixturing, cost, its influence on electrical resistivity and its relation to diameter based on buckling considerations. The diameter of the specimen is based on the raw material size, its relation to electrical resistivity and the fastener size in the fixtures.

Figures 13, 14 and 15 show the specimen design used in the current experiments. Fig. 13 shows a specimen that can be mounted in the hot jaws directly. This design permits accurate alignment of the specimen halves and the utilization of a steep thermal gradient between the bond interface end and the supported end of the specimen. This design, however, requires a longer specimen. Fig. 14 shows the specimen design that permits the usage of a shorter length specimen with the help of a stainless steel transfer fixture.
Fig. 13  
(a) Direct hot jaw specimen with metric threads, Design I;  
(b) Hot jaw specimen with a reduced length of smaller diameter, Design II
Fig. 14 (a) Specimen Design III, used with the stainless steel fixture; and (b) specimen Design IV, used with the stainless steel fixture.
Fig. 15 (a) Specimen Design V, used with the stainless steel fixture and (b) Specimen Design VI, used with the stainless steel fixture.
The problems to be anticipated from this design include shallow temperature gradients (the specimen is no longer directly in contact with the water cooled hot jaws) and slight misalignment due to the additional threaded sub assembly incorporated. The design in Fig. 14 (b) shows a specimen design with an interface diameter less than the minor diameter of the thread. Fig.16 shows further variations in specimen design that permit an average interface diameter when compared to Fig.14 (a) and (b) and an additional design which permits longer bonding times due to the provision of an increased length.

Fig. 16 (a) shows a 25 Vol % SiCp/6061 Al MMC machined sample ready to be mounted in the stainless steel hot jaws directly. One end of the bar was threaded to M 10 x 1.5. Fig. 16 (b) shows the composite material mounted directly in the hot jaws. Both these figures also show the pre-welded thermocouple (chromel/alumel) junction attached to the specimen (after being located in a drilled hole) with a ceramic paste.

4.4.4 BASE METAL CHARACTERIZATION (THERMO-MECHANICAL SIMULATION):

The purpose of this simulation was to impose i) a thermal and ii) a thermo-mechanical cycle on the specimen similar to that which the bonded specimen subsequently undergoes and thereby understand the effects of the bonding thermo-mechanical cycle on the base metal. It is necessary to carry out these tests in view of the fact that very little material property data is available for the metal matrix composites. Even in those cases where some property data is available, information regarding high temperature properties and related microstructural effects could not be found in the search carried out in this study.
Fig. 16 25 Vol% SiC\textsubscript{p}/6061 Al MMC 3/8" diameter bar, (a) machined and ready to be bonded and (b) mounted in the stainless steel hot jaws directly. Note the ceramic adhesive locating the thermocouple wires.
In all the simulation studies the 25 VOI % SiCp/6061 Al MMC materials (0.55 " diameter extrusion) were used.

This simulation was divided into three parts:

i) simulation utilizing a furnace heat treatment cycle i.e., the specimen undergoes the thermal cycle only; cycled and followed by post heat treatment,

ii) simulation where the specimen undergoes the thermo-mechanical cycle on the Gleeble 1500 system followed by a post cycle heat treatment and analyzed by tension testing and examination of the fracture surfaces by the scanning electron microscope and

iii) Gleeble thermo-mechanical simulation followed by post cycle heat treatment and analysis by microhardness evaluation and examination of the optical microstructure.

4.4.4.1 THERMAL CYCLE SIMULATION (IN HEAT TREATMENT FURNACE):

This process utilizes a regular heat treatment furnace to impose the desired thermal cycles on the specimen. The specimen is introduced into a preheated furnace with the desired temperature maintained and soaked for varying times followed by air cooling. Thereafter one-half of the samples were subjected to a resolutionizing and an aging treatment. Temperature measurement (+ or - 3 ° C) was accomplished either through a thermocouple system attached to a sample or a thermocouple placed along with the components being thermally cycled or heat treated. It took about 3 to 4 minutes for the
sample to reach the soak temperature. Specimen were thermally cycled to various
temperatures from 500 to 590 °C, and for times ranging from 5 minutes to 90 minutes.
The typical sample sizes used for these experiments were 0.55 " in diameter and 0.2 to
0.25 " in length. After the thermal cycling, the samples were cut, mounted in bakelite and
polished. They were then subjected to microhardness testing and examination with
optical microscopy.

4.4.4.2 SIMULATION OF THERMO-MECHANICAL CYCLES USING THE
 GLEEBLE SYSTEM:

These simulation experiments were performed on the Gleeble 1500 system, and
were divided into two parts. Fig. 17 shows the sample design used in these
experiments.

The first batch of Gleeble simulated samples were thermally cycled at temperatures
of 540, 565 and 575 °C for 30 minutes in all cases and an additional sample for a time of
10 minutes for the 575 °C case. All these samples were subjected to a compressive
pressure during the thermo-mechanical cycle similar to that experienced during actual
bonding. Fig. 17 (a) shows the sample design used in these cases. Fig. 17 (b)
represents a sample which was held in the stainless steel hot jaws with the stainless steel
fixture, similar to that shown in Fig. 12. These second batch of samples were then
subjected to thermo-mechanical cycles with temperatures ranging from 540 to 580 °C
and times ranging from 5 minutes to 60 minutes.
Fig. 17  Gleeble samples for Thermo-mechanical simulation
(a) for strength and fracture analysis (on SEM) and
(b) for microhardness and optical micrography.
They were heated and cooled in the same fashion as were the samples to be bonded. During the study, a partial vacuum of 3.5 milli-torr was maintained in the specimen chamber. The specimen were subjected to a compressive pressure during the simulation. The deformation experienced by the samples, calculated as the variation in the maximum diameter (measured after thermo-mechanical cycling) to the original diameter as a ratio of the original diameter was calculated in each case.

4.4.4.3 POST CYCLE HEAT TREATMENT:

Two schedules of heat treatment were used for heat treating the base metal in this study. Both the schedules involved solution treatment at 530 °C, water quenching and room temperature aging for 48 hours. Subsequently, the specimen were artificially aged at 175 °C, for 2 hours as per schedule # 1 or for 3 hours as per schedule # 2. Schedule # 1 was used for heat treatment of the samples exposed to the thermal and thermo-mechanical simulation experiments conducted in this study.

To optimize the heat treatment schedules for the bonded samples, two samples of the base metal were also artificially aged after solution treatment and naturally aged as above for 7 and 13 hours.

4.4.4.4 SAMPLE MECHANICAL TESTING:

The first batch of the samples Gleeble simulated as indicated in section 4.4.4.2, were machined to tensile specimen per ASTM E - 8 112 and tensile tested as per standard ASTM metallic materials testing practice at a displacement rate of 0.01 " per minute.
After the simulation cycle, a second batch of the Gleeble simulated samples were cut with a diamond saw in the location of the thermocouple, mounted in epoxy, polished and etched which were subsequently subjected to microhardness testing and optical microscopy.

4.4.5 DIFFERENTIAL SCANNING CALORIMETRY

Differential scanning calorimetry (DSC) studies were performed for the 25 Vol % SiC<sub>p</sub>/6061 Al MMC extruded bar stock of 0.55" diameter on a Perkin-Elmer DSC-7 apparatus in order to observe the solidus and liquidus temperatures of the composite material. Even though theoretically adding silicon carbide particulate reinforcement to the 6061 aluminum matrix should not affect the solidus and liquidus temperatures of the composite, this study was however undertaken to confirm this assumption. Test were performed on as received material and on heat treated base metal samples to see any changes resulting from the heat treatment.

4.4.6 LIQUID PHASE DIFFUSION BONDING

4.4.6.1 SPECIMEN CLEANING PROCEDURES

The specimen to be bonded after machining were normally subjected to cleaning with one of the following procedures:

i) **Procedure I**

Chromic acid 6 to 12 oz,
Sulfuric acid 19 to 24 fluid oz, 
water 1 gal, 
temperature of bath 43 to 82 °C and 
Immersion time up to 20 min.

ii) Procedure II
Nitric acid 64 to 96 fluid oz, 
Hydrofluoric acid 3 to 24 fluid oz, 
water 1 gal, 
Bath temperature Room temperature and 
Immersion time 1 to 5 min.

iii) Procedure III
Electro-polishing in a solution of 25 Vol% nitric acid and 75 Vol% methyl alchohol at room temperature for times ranging from 15 seconds to 120 seconds and at 5 volts.

4.4.6.2 SURFACE FINISH:

The types of surface finish employed for this study include "i) as turned surfaces, ii) turned and wire brushed surfaces (with a stainless steel wire brush) and iii) turned and electropolished surfaces", at the faying interface. The former two methods were employed before chemical cleaning and the latter was employed in place of chemical cleaning.
4.4.6.3 **BONDING ON THE GLEEBLE 1500**

Specimen of the designs displayed in Figs. 14 through 16 were subjected to Gleeble bonding cycles within 24 hours after chemical cleaning. Temperature measurement was through a chromel/alumel thermocouple junction percussion welded to the sample 2 mm from the interface. (In a few cases pre-welded chromel/alumel thermocouples, bonded to the specimen surface with a ceramic adhesive, were used). Attainment of a good thermocouple bond to the sample was very important for these experiments in view of the elevated temperatures used. In quite a few cases, some thermocouples came off the samples at 550 °C. It appeared that an improperly bonded thermocouple was unable to sustain the thermal stresses at these temperatures. The thermocouple bond was checked with manual pull-off tests before the sample was assembled into the Gleeble. The force and temperature were monitored and recorded during the experiment on a strip-chart recorder.

Before initiation of the experiment, the machine force controller was checked for zero force in the free condition. The sample was then loaded into the machine and the computer controlled thermo-mechanical cycle was initiated. During this stage the specimen was held at the bonding temperature for the programmed time with the desired compressive loading. The specimen chamber was maintained at a vacuum of $1 \times 10^{-4}$ torr or better. After the bonding cycle, the specimens were cooled in vacuum to a temperature of about 100 °C. Most of the bonded samples were subsequently heat treated as per Schedule # 1 or 2 (listed in Section 4.4.4.3). They were then either mounted for metallography or were machined for tension testing.
4.4.7 METALLOGRAPHY PROCEDURES

The samples were cut with a Buehler diamond saw and the longitudinal sections were mounted in Bakelite. In the case of the Gleeble thermally simulated samples, the transverse sections at the thermocouple locations were mounted. The following procedures were used to polish the specimen for microstructural examination:

METHOD I:

i) grinding with a 600 grit silicon carbide paper,
ii) polishing with diamond paste of size 9 and 3 microns,
iii) polishing with 1 micron diamond suspension,
iv) polishing with 0.3 micron alumina,
v) polishing with the colloidal silica suspension
vi) etching with Keller’s etch.

Cleaning in water was employed where necessary and ultrasonic cleaning in methyl alcohol was used between each of the above steps.

METHOD II:

i) grinding with a 600 grit silicon carbide paper,
ii) polishing with 9 micron and 3 micron diamond paste,
iii) polishing with 0.3 micron alumina
iv) final polishing with Magomet.

Cleaning in water was employed where necessary and ultrasonic cleaning in methyl alcohol was used between each of the above steps.
METHOD III:

Same as above except the elimination of step iii) above.

METHOD IV:

Polishing with 9, 3 and 1 micron diamond paste with ultrasonic cleaning in methyl alcohol in between, making sure that water was not used.

Methods I and II were employed for preparing bonded specimen for characterization. Step III and IV were used for evaluating the thermally and thermo-mechanically simulated samples. Selecting metallographic polishing techniques for these materials was not easy as specific literature information is scarcely available and this necessitated the use of modifications as work progressed in order to improve the details.

4.4.8 MICROSTRUCTURAL CHARACTERIZATION

The microstructural characterization of the polished samples was achieved with the following techniques: 1) optical microscopy at a magnifications of 50, 100, 200 and 400 X; 2) scanning electron microscopy (SEM) using a HITACHI S-510 to characterize the polished samples as well as the fracture surfaces; 3) SEM-Energy dispersive X-Ray microanalysis (SEM-EDAX) using a JEOL 35A equipment to obtain the chemistry of the desired regions; 4) electron probe microanalysis (EPMA) on a CAMECA SX 50. Both optical microscopy and the scanning electron microscopy were employed to obtain information at the bond interface and the material to either side of the interface. Attempts were made to see if any reinforcement-matrix debonding had occurred due to the
bonding thermo-mechanical cycle. With the help of microstructural and other information, the processes in the bonding were analyzed. SEM-EDAX was used to identify the chemistry gradients near the interface. EPMA was also used to obtain information on the chemistry of the sample in a direction transverse to the bonding interface. Also, chemistry profiles of single elements were obtained.

4.4.9 EVALUATION OF BOND PROPERTIES

The bond mechanical properties were evaluated as follows: 1) the microhardness testing was done with a Buehler microhardness tester; 2) the Knoop hardness values were determined at a load of 100 grams; 3) tension testing was performed at the Edison Welding Institute (EWI), at a displacement rate of 0.01" per minute as per ASTM E-8, Tension Testing of Metallic Materials. From the above test, percentage elongation values were also obtained; 4) a limited amount of bend testing was done at the EWI.
CHAPTER V

5.0.0 RESULTS

5.1.0 BASE MATERIAL HEAT TREATMENT

Two schedules of heat treatment were used for heat treating the base metal in this study. Both the schedules involved solution treatment at 530 °C, water quenching and room temperature aging for 48 hours. Subsequently, the specimens were artificially aged at 175 °C for 2 hours as per schedule # 1 and for 3 hours as per schedule # 2. In addition, two more base material samples were solution treated, water quenched, naturally aged for 48 hours and artificially aged for 7 and 13 hours respectively. Fig. 18 shows the variation of microhardness with the aging time at 175 °C, as determined in this study.
Fig. 18 Variation of base material (25 Vol% SiCp/6061 Al MMC) microhardness with artificial aging time (the hardness value of 120 shown at zero time is that of the material in the as fabricated condition).
5.2.0 DIFFERENTIAL SCANNING CALORIMETRY

Differential scanning calorimetry (DSC) studies were performed on Perkin-Elmer DSC-7 equipment to determine the changes in the solidus temperature of the matrix alloy. It might be expected that there is no change in the solidus temperature of the matrix alloy due to the thermo-mechanical processing required in the manufacture of the composite. In order to confirm this, the DSC studies were performed.

The results of the first study are presented in Figures 19 through 21. A 25 Vol% SiCp/6061 Al composite sample weighing 15.16 milligrams was heated from 500 to 650 °C at a rate of 10 °C per minute, held at 650 °C for 20 minutes and was cooled to 450 °C at a rate of 10 °C per minute. Fig. 19 shows the DSC trace on heating and Fig. 20 shows the DSC trace on cooling. Fig. 21 provides a time basis of the heating and holding cycle. Fig. 22 illustrates the trace of another 22.38 milligram sample heated from 500 to 580 °C at a rate of 10 °C per minute and held at 580 °C for 60 minutes.

Figures 23 through 25 provide the DSC traces of another sample weighing 44.3 milligrams, in the heat treated condition (solution treated at 530 °C, water quenched, naturally aged at room temperature for 48 hours and artificially aged at 175 °C for 13 hours). Fig. 23 provides the time base trace of the heating from 500 to 670 °C at a heating rate of 10 °C per minute and isothermal holding at 670 °C for 10 minutes. Fig. 24 provides the DSC trace of holding at 670 °C for 10 minutes and cooling from 670 to 450 °C at rate of 10 °C per minute. Fig. 25 shows the heating trace of the above sample, heated subsequently to 670 °C after cooling to 450 °C.
Fig. 19 DSC trace (on heating) of a 25 Vol % SiCp/6061 Al MMC at a heating rate of 10 °C per minute. Note the solidus temperature of 585 °C.
Fig. 20 DSC trace (on cooling) of a 25 Vol % SiCp/6061 Al MMC at a cooling rate of 10°C per minute. Note the approximate indication of the solidus temperature at 585°C.
Fig. 21  DSC trace (time base) of heating to and holding for 20 minutes at 650 °C of a 25 Vol % SiCp/6061 Al MMC at a heating rate of 10 °C per minute. Note heat absorption indicating the probable formation of aluminum carbide due to the reaction of silicon carbide and liquid aluminum.
Fig. 22  DSC trace (time base) of heating at a rate of 10 °C per minute to and holding for 60 minutes at 580 °C of a 25 Vol % SiCp/6061 Al MMC. Note no major heat absorption or rejection.
Fig. 23 DSC trace (time base) of heating at a rate of 10 °C per minute from 500 to 670 °C and holding for 10 minutes of a 25 Vol % SiCp/6061 Al MMC at a heating rate of 10 °C per minute. Note very little heat absorption to indicate the probable formation of aluminum carbide due to the reaction of silicon carbide and liquid aluminum, unlike that seen in Fig. 21.
Fig. 24  DSC trace of holding at 670 °C for 10 minutes and cooling at a rate of 10 °C per minute from 670 to 450 °C of a 25 Vol % SiCp/6061 Al MMC. Note the solidus at approximately 580 °C.
Fig. 25  DSC trace of heating from 450 °C at a rate of 10 °C per minute to 670 °C, of a 25 Vol % SiCp/6061 Al MMC (reheating the same sample of Fig. 24). Note the secondary heat absorption at around 557 °C.
5.3.0 THERMAL SIMULATION STUDIES

5.3.1 THERMAL SIMULATION IN FURNACE

In the ingot metallurgy 6061Al, elevated temperature processing can cause precipitate dissolution or coarsening depending on the time and temperature employed. These can correspondingly result in very different mechanical properties. Consequently, any elevated temperature thermal processing must take these changes into account.

Samples from the SiCp/6061 Al MMC (0.55" diameter bar stock) of 0.25" thickness were thermally cycled to simulate the thermal excursions experienced in the liquid phase diffusion bonding process. The experiments were carried out in a heat treatment furnace with the procedure as described in the section 4.4.4.1. After heat treatment, samples were cut with a diamond saw, mounted in Bakelite and polished according to Method II as indicated in the section 4.4.7. Figures 26 and 27 show the variation of microhardness values obtained from these thermal simulation experiments with temperatures ranging from 500 to 590 °C and times from 5 to 90 minutes. Fig. 26 shows the results for the furnace simulated base metal and Fig. 27 shows the results for the furnace simulated and post-simulation heat treated base metal. The type of heat treatment employed for this material is as given below:

i) Solution treatment at 530 °C for one hour followed by water quenching,
ii) Natural aging at 25 °C for 48 hours and
iii) Artificial aging at 175 °C for 2 hours and air cooling from the aging temperature.
Fig. 26 Variation of hardness in the base metal after furnace simulation.
Fig. 27 Variation of hardness in the base metal after furnace simulation and post-simulation heat treatment as per schedule #1.
5.3.2 **THERMO-MECHANICAL SIMULATION WITH GLEEBLE 1500**
(MICROHARDNESS AND OPTICAL MICROSTRUCTURE EVALUATION):

Table. 2 and Fig. 28 show the results of the thermo-mechanical simulation experiment on the Gleeble 1500 system with the SiCp/6061 Al composite material. Table. 2 provides the loading conditions and the resulting deformation in the samples which were thermally cycled from 540 to 580 °C for 5 to 60 minutes.

5.3.3 **THERMO-MECHANICAL SIMULATION WITH THE GLEEBLE**
(ANALYSIS OF STRENGTH AND FRACTURE BEHAVIOR):

In the conventional ingot metallurgy alloys, the purpose of thermo-mechanical simulation would be to study the effects of the simulation process on the microstructure and related mechanical properties. This could take place from the various metallurgical processes occurring and essentially depends on the simulation temperature and loading with reference to the critical material temperatures and the load carrying capacity of the material at the temperature concerned.

Table. 3 summarizes the mechanical test results for the base material and the thermo-mechanical simulation study samples (using the Gleeble 1500 system) of the 25 Vol % SiCp/6061 Al MMC material (0.55” diameter extrusion). All the samples that were thermo-mechanically simulated were subsequently heat treated to the schedule as shown.
Table 2: Results of Gleeble thermo-mechanical simulation (Microhardness and optical microstructure analysis).

<table>
<thead>
<tr>
<th>#</th>
<th>Simulation Conditions</th>
<th>Deformation</th>
<th>Heat Treat</th>
<th>Knoop Hardness (100 gm load)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temp deg C</td>
<td>Time min</td>
<td>Comprn. Pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.</td>
<td>540</td>
<td>5</td>
<td>870 psi</td>
<td>1.8%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>2.</td>
<td>540</td>
<td>30</td>
<td>914 psi</td>
<td>1.8%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>3.</td>
<td>540</td>
<td>60</td>
<td>870 psi</td>
<td>1.8%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>4.</td>
<td>570</td>
<td>5</td>
<td>706 psi</td>
<td>4.3%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>5.</td>
<td>570</td>
<td>30</td>
<td>706 psi</td>
<td>3.9%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>6.</td>
<td>570</td>
<td>60</td>
<td>716 psi</td>
<td>6.43%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>7.</td>
<td>580</td>
<td>5</td>
<td>352 psi</td>
<td>4.07%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>8.</td>
<td>580</td>
<td>30</td>
<td>650 psi</td>
<td>10.7%</td>
<td>Sch. #2</td>
</tr>
<tr>
<td>9.</td>
<td>580</td>
<td>60</td>
<td>616 psi</td>
<td>11.9%</td>
<td>Sch. #2</td>
</tr>
</tbody>
</table>

Sch #2: Solution treat at 530 °C for one hour, water quench; 25 °C for 48 hours; Age 175 °C for 3 hours and air cool.
Fig. 28 Microhardness data for Gleeble thermo-mechanical simulated 25 Vol% SiCp/6061 Al composite material (at a partial vacuum of 3.5 milli-torr).
Table 3. Thermo-mechanical simulation results for the base metal on the Gleeble (Results of mechanical testing)

<table>
<thead>
<tr>
<th>#</th>
<th>Simulation Conditions</th>
<th>Defor-Mation</th>
<th>Heat Treat Condition</th>
<th>Mech Test Ten.str % Elongn.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.B.M</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
</tr>
<tr>
<td>2.B.M</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
</tr>
<tr>
<td>3.B.M.</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
</tr>
<tr>
<td>4.</td>
<td>30 min.</td>
<td>540°C</td>
<td>-730 psi</td>
<td>1.29 %</td>
</tr>
<tr>
<td>5.</td>
<td>30 min.</td>
<td>565°C</td>
<td>-580 psi</td>
<td>6.45 %</td>
</tr>
<tr>
<td>6.</td>
<td>30 min.</td>
<td>575°C</td>
<td>-730 psi</td>
<td>16.13 %</td>
</tr>
<tr>
<td>7.</td>
<td>10 min.</td>
<td>575°C</td>
<td>-260 psi</td>
<td>6%</td>
</tr>
</tbody>
</table>

Samples # 2 to 7 were water quenched after solution treatment at 530 °C.
B.M : 25 Vol % SiCp/6061 Al MMC (0.55 " diameter extrusion);
R.T : Room temperature.
The heat treatment schedule # 2 used in this study is as given below:

i) solution treatment at 530 °C for one hour and water quenching;

ii) natural aging at 25 °C for 48 hours;

iii) artificial aging at 175 °C for 3 hours and air cooling from the aging temperature.

The results of mechanical testing have been presented in Table 3. One of the heat treated base materials failed in the jaws at a tensile strength of 69.1 ksi. This is nearer to the ultimate tensile strength values of 68.1 to 75 ksi reported in the literature as indicated in the Table. 1 in the second chapter. Considering this as the base, it can be observed that thermo - mechanical treatment of 30 minutes at a compressive loading of 730 psi (resulting in a deformation of 1.29 %) at 540 °C, has virtually no effect on the tensile properties (retains 93 % of base metal value). Thermo - mechanical simulation at 565 °C for 30 minutes at a compressive loading of 580 psi (resulting in a deformation of 6.45 %) yields a joint strength of 46.49 ksi (67.3 % of base metal value), whereas a loading of 730 psi (deformation of 16.13 %) for 30 minutes at a temperature of 575 °C results in a tensile strength of 43.2 ksi (62.5 % of base metal value). One surprising result of this experiment is the value obtained for a treatment time of 10 minutes at the temperature of 575 °C. A tensile strength of 60.1 ksi (86.9 % of base metal value) at a compressive loading of 260 psi (deformation of 6 %) has been obtained. These results have to be analyzed along with those of the furnace and thermal simulation studies reported in the earlier sections.
Figures 29 to 34 show the microstructural features of the fracture surfaces of the tensile tested Gleeble thermo-mechanical simulated and heat treated samples as presented in the Table 3. Figures 29 (a) through (d) show the fracture surfaces of the sample thermo-mechanically treated at 540 °C. Fig. 29 (a) shows the macroscopic features of fracture. Fig. 29 (b) shows the fracture surface near the outer edge. Figs. 29 (c) and (d) show the details of the features of the matching half of the fracture surface. Figures 30 (a) through (d) show the tensile tested fracture surfaces of samples thermo-mechanically treated at 565 °C, for 30 minutes, at a compressive loading of 580 psi (resulting in a deformation of 6.45 %). Figs. 30 (c) and (d) show the fracture surfaces of the second half of the sample. Fig. 30 (c) shows dimples and voids. Compared to the samples thermally treated at 540 °C, an increase in void density may be observed. Fig. 30 (d) shows an enlarged view of the fracture surface at the central portion of Fig. 30 (c).

Figs. 31 (a) through (f) show the fracture surface features of a sample thermo-mechanically treated at 575 °C for 30 minutes, at a compressive loading of 730 psi (deformation of 16.13 %). Fig. 31 (a) shows a low magnification macroscopic view of the fracture surface. An enlarged view of the fracture surface in Fig. 31 (b) shows larger dimples and voids. Fig. 31 (c) shows the features near a crack opening on the second half of the fracture surface of the sample. Very clear evidence of fibrous fracture, including the formation and orientation of the fibrous material may be seen. Fig. 31 (d) shows the fibrous material at another crack mouth at low magnification. In both these figures, the fibrous material being formed and already formed (just behind the immediate top surface) may be seen.
Fig. 29  Fracture surface of sample thermo-mechanical treated at 540 °C for 30 min at 730 psi compressive loading (deformation 1.29%); (a) low magnification showing the fracture pattern; (b) shows the stepped fracture and dimples.
Fig. 29  Fracture surface of sample thermo-mechanical treated at 540 °C for 30 min at 730 psi compressive loading (deformation 1.29 %); (c) showing voids, dimples and particulate fracture and (d) showing the particulate located in dimples, at arrows (Notice lack of interfacial decohesion).
Fig. 30 Fracture surface of sample thermo-mechanical treated at 565 °C for 30 min at 580 psi compressive loading (deformation 6.45%); (a) low magnification showing stepped or terraced fracture; (b) fibrous fracture surface near a crack mouth.
Fig. 30 Fracture surface (second half) of sample thermo-mechanical treated at 565 °C for 30 min at 580 psi compressive loading (deformation 6.45%), (c) dimples and voids; (d) particulates at the base of dimples (at arrow) in an enlarged view of the central region of the above figure. Note no debonds and similarities to Fig. 29 (d).
Fig. 31 Fracture surface of sample thermo-mechanical treated at 575 °C for 30 min at 730 psi compressive loading (deformation 16.13 %); (a) at low magnification shows the macroscopic view of the fracture surface; (b) shows large voids (compared to Fig. 30 (c)) and dimples.
Fracture surface of a sample thermomechanically treated at 575 °C for 30 min at 730 psi compressive loading (at a deformation of 16.13%); (c) at low magnification shows fibrous fracture near a crack; (d) shows fibrous fracture features near and inside a crack mouth.
Fig. 31 Fracture surface of a sample thermo-mechanical treated at 575 °C for 30 min at 730 psi compressive loading (at a deformation of 16.13 %); (e) shows the orientation and morphology of the fibrous features; (f) shows a close view of the fibrous features. Note that it is not possible to discern clearly the association of particulates with the fibrous features.
It is expected that the fibrous material is being formed due to the ductility of the aluminum matrix. Figs. 31 (e) and (f) show the enlarged view of the fibrous material. In Fig. 31 (e) and (f), the fibrous material protruding from the fracture surface can be seen.

Figs. 32 (a) through (f) show the fracture surface features of a sample thermomechanically treated at 575 °C for 10 minutes at a compressive loading of 260 psi (a deformation of 6%). This sample was the only one held in stainless steel inserts in a set of copper jaws. Fig. 32 (a) shows the fracture surface at a low magnification. Fig. 32 (b) shows an enlarged view of the central area shown in Fig. 32 (a). Fig. 32 (c) shows a view near a crack opening. Fig. 32 (d) shows an enlarged view of the particulate fracture features seen in Fig. 32 (c). Fig. 32 (e) shows a close view of the fibrous material. Fig. 32 (f) shows dimples indicating ductility and particulate material associated with the dimples.

5.4.0 LIQUID PHASE DIFFUSION BONDING

5.4.1 AL-SI-MG INTERLAYER

In order to conserve costly MMC materials and to establish bonding boundary conditions, initial trials of the liquid phase diffusion bonding were conducted on 0.1" thick 6061-T6 aluminum alloy sheet (without SiC reinforcement) in a lap joint configuration. The specimen were cut to 1.5" x 0.375" size and were cleaned using the procedure #1 outlined in the section 4.4.6.1. They were held in special stainless steel fixtures in the copper jaws.
Fig. 32 Fracture surface of a sample thermo-mechanical treated at 575 °C for 10 min at 260 psi compressive loading (a deformation 6%); (a) at low magnification shows dimples and voids; (b) shows dimples, voids and particulates.
Fig. 32 Fracture surface of a sample thermo-mechanically treated at 575 °C for 10 min at 260 psi compressive loading (deformation of 6%); (c) at low magnification shows formation of fibrous fracture features near a crack mouth (at arrow); (d) an enlarged view of the above figure shows particulate fracture (at arrow).
Fig. 32 Fracture surface of a sample thermo-mechanical treated at 575 °C for 10 min at 260 psi compressive loading (deformation of 6%); (e) & (f) show fiber morphology (at A) and fracture suggesting dimples and particulates associated with dimples (at B). Note similarities to Figs. 29(d) and 30(d).
Temperature control was achieved through Platinum/Platinum-13% Rhodium thermocouple wires percussion welded to the sample (further experimentation indicated that the Platinum/Platinum-13% Rhodium is not a reliable system for temperature measurement in the aluminum base alloys at the elevated temperatures employed). The specimens were bonded at 575 °C for one hour, in rough vacuum (5 milli-torr) with the thinned Al-Si-Mg interlayer (whose chemistry and melting point are provided later in this section) with a lap width of 0.5 ".

The interlayer, which as received was about 0.010 to 0.013 " thick, was thinned to about 0.004 to 0.005 " thickness by polishing on a 600 grit silicon carbide paper. Fig. 33 (a) shows a sample in a transverse cross section at low magnification, indicating the remnants of the foil. Fig. 33 (b) shows another sample where the interlayer was completely diffused away also indicating the presence of the diffusion zone. Fig. 34 shows the differential thermal analysis of the Al-Mg-Si interlayer foil. It indicates two heat absorption steps, the primary initiating at 595.8 °C and the secondary corresponding to the ternary eutectic solidification. Fig. 35 reveals the remnants of the interlayer foil at the bond location.

In section 2.3.8.3, the time for isothermal solidification with the Al-Si-Mg interlayer was calculated. Considering the the diffusion coefficient of silicon in aluminum at 575 °C as 9.97 X 10^-9 cm^2/sec \(^{117}\), constant K as 1 and time \(t\) as 3600 seconds, the diffusion distance can be calculated from:

\[
X = K (Dt)^{1/2}, \quad (23)
\]
Fig. 33 Micrograph of a 6061 Al sheet liquid phase bonded with an Al-Si-Mg interlayer, (a) transverse cross-section showing remaining interlayer and continuity of grains on either side of the interlayer (as shown by arrows) and (b) a diffusion zone (bound by A - A) showing complete dispersal of the interlayer.
Fig. 34  DTA trace of cooling from 650 °C at a rate of 10 °C per minute to 450 °C, of the Al-Si-Mg interlayer foil. Note the onset of a minor solidification product at 595.8 °C.
Fig. 35 Low magnification Micrograph of a 6061 Al sheet liquid phase bonded with an Al-Si-Mg interlayer, transverse cross-section showing remaining interlayer.
The diffusion coefficient indicated above compares favorably with the work of Fujikawa et.al., who provided the following equation for the temperature dependence of the impurity diffusion coefficient of silicon in aluminum:

$$D_{Si/Al} = 2.02^{+0.97}_{-0.66} \times 10^{-4} \exp\{-\left[(136\pm3)kJ \text{ mol}^{-1}/RT\right]\} m^2/s \ (24)$$

The above equation provides the value of $9.68 \times 10^{-9} \text{ cm}^2/\text{sec}$ for $D_{Si/Al}$ at 575 °C. The diffusion distance calculated from equation (23) is 60 microns, which compares favorably with that experimentally observed.

The following observations were made based on this study:

i) Continuity of grain boundaries across where the interface has been indicated the occurrence of isothermal solidification,

ii) The thickness of the diffusion zone corresponded to the calculated diffusion distance.

Several difficulties were experienced with this interlayer, in spite of the confirmation of the liquid phase bonding process as explained above. They were:

i) as shown in the section 2.3.8.3, an interlayer of thickness 0.004 " can not be completely diffused away in the temperature/time conditions used in this experiment. Time much longer than one hour would be required for complete diffusion and was not considered since the low resistance of aluminum in the resistance heating method employed would impose an undue thermal load on the machine and thus several hours times were considered impractical for most bonding applications; ii) thinning the
interlayer down to a thin uniform thickness was not practicable due to the brittleness of the interlayer and iii) results of the differential thermal analysis (DTA) as shown in Fig. 34 combined with the chemical analysis of the foil (Aluminum 75%, Magnesium 4.46% and silicon 18.45%) indicated that it will be impossible to bring the interlayer completely to a molten state unless much higher temperatures (> 596 °C) were used (Fig. 35 revealed this). Higher temperatures were not considered as the solidus temperature of the alloy would be exceeded. The next logical step would be the selection of an interlayer which can overcome the above problems.

5.4.2 SILVER INTERLAYER

A silver interlayer of 99.9% purity, of 0.001" thickness obtained from Johnson Matthey, Seabrook, New Hampshire, was considered to satisfy most of the above requirements as already explained in the section 2.3.8.4. This interlayer provided the necessary ductility and forms a low melting eutectic liquid with aluminum at 567 °C. However the 0.001" thickness was the smallest thickness commercially available.

5.4.3 LIQUID PHASE DIFFUSION BONDING WITH THE SILVER INTERLAYER:

The results of the thermal and thermo-mechanical simulation studies presented in Section 5.3.0 have restricted the bonding temperatures to range from 567 to 580 °C and bonding times from 10 to 30 minutes, with preferably the longer times used at the lower temperatures. As shown in the previous section, in order to completely isothermally solidify the 0.001" thickness silver interlayer, longer times than are permitted by the
thermal simulation are necessary. To satisfy these conflicting requirements, it has been decided that the thermal simulation restrictions be maintained and an apparent interlayer thickness reduction be achieved through the removal of excess liquid by extrusion.

5.4.3.1 LIQUID PHASE BONDING OF 6061 ALUMINUM MATERIAL:

Once again to conserve expensive MMC materials, the initial trials of liquid phase bonding with the silver interlayer were conducted with the Al - 6061 alloy. Fig. 36 shows a 6061 Al rod, 3/8" diameter, liquid phase diffusion bonded from two 1.5" length rods, with the 0.001" thick silver interlayer, for 5 minutes at 570 °C in a partial vacuum of 5 milli-torr and under a compressive load of 400 psi. The sample was bonded in the stainless steel hot jaws directly. An upset at the interface and the occurrence of cracks near the interface may be noted. Fig. 37 shows another Al - 6061 rod bonded for 10 minutes. Formation of fibrous material at the interface due to the extrusion may be observed. It can be concluded that during bonding, the 6061 aluminum alloy could not tolerate the thermo-mechanical loading resulting from the bonding cycle. At shorter bonding times cracking occurred and at longer times severe fibrous extrusion was observed.

5.4.4 LIQUID PHASE DIFFUSION BONDING OF THE COMPOSITE MATERIAL:

Liquid phase diffusion bonding trials were initiated on a 0.37" diameter extruded bar stock of the 25 Vol % SiCp/6061 Al MMC material.
Fig. 36 A liquid phase diffusion bond in a 3/8" diameter 6061 Al bar. The bond was obtained at 570 °C for 5 minutes with a 0.001" silver interlayer at 5 milli-torr vacuum and a compressive load of 400 psi. The upset and cracking at the interface may be noted.
Fig. 37 A liquid phase diffusion bond in a 3/8" diameter 6061 Al bar. The bond was obtained at 570 °C for 10 minutes with a 0.001" silver interlayer at 5 milli-torr vacuum and a compressive load of 400 psi. Note the fibrous extrusion (at arrow).
The specimens, after machining and cleaning in acetone were treated with cleaning procedure #2 (section 4.4.6.1), to minimize the oxide layer thickness. Fig. 38 shows the bonded composite material. The liquid phase bonding was carried out at 570 °C for 10 minutes, with an 0.001 " silver interlayer and a 300 psi compressive load, at 5 milli-torr vacuum at the bonding temperature. The excess of the eutectic liquid was extruded out. The deformation at the interface of the bonded sample was 27.6%. A comparison of Figs. 36 and 37 with Fig. 38 reveals that there was no external damage to the composite material, whereas the 6061 Al had undergone severe fibrous extrusion. The deformation in the composite material, was manifested as an upset at the interface. Fig. 39 shows another sample of the composite material liquid phase diffusion bonded at 570 °C for 10 minutes with an 0.001 " silver interlayer at 5 milli-torr vacuum and 300 psi compression. The deformation at the interface in this case was 44%. The material had undergone the deformation without showing any fibrous extrusion externally. Fig. 40 shows another sample which was liquid phase diffusion bonded at 570 °C for 10 minutes with an 0.001 " silver interlayer at 5 milli-torr vacuum and a 300 psi compressive load. Part of the eutectic liquid was extruded out. The deformation at the interface was 41%.

5.4.5 MACRO & MICROSTRUCTURAL CHARACTERIZATION:

5.4.5.1 MACROSTRUCTURE:

Figs. 41 and 42 show the macrographs of 0.37 " dia 25 Vol% SiCp/ 6061 Al MMC bars liquid phase diffusion bonded with a 0.001 " silver interlayer and sectioned longitudinally.
Fig. 38  25 Vol% SiCp/6061 Al MMC 3/8 " diameter bars liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 ° C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi. The resulting deformation at the interface is 27.6%. Note the spectacular change in the material behavior as compared to Figs. 36 and 37.
25 Vol% SiCp/6061 Al MMC 3/8" diameter bars liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi. The resulting deformation at the interface is 44%.
Fig. 40 25 Vol% SiCp/6061 Al MMC 3/8 " diameter bars liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi. The resulting deformation at the interface is 41 %.
The sample in Fig. 41(a) was bonded at 570 °C for 10 minutes at 5 milli-torr vacuum with a compressive load of 300 psi and a deformation of 27.6%. The sample in Fig. 41(b) was bonded at 580 °C for 20 minutes at 5 milli-torr vacuum with a compressive load of 550 psi and a deformation of 35%. The sample in Fig. 42(a) was bonded at 580 °C for 20 minutes at 400 milli-torr vacuum and a compressive load of 270 psi with a resulting interfacial deformation of 3%. The sample in Fig. 42(b) was bonded at a temperature of 580 °C for 20 minutes with 300 psi compressive pressure and at a vacuum of 5 x 10⁻⁵ torr and 5% deformation. Figs. 41(a) and (b) represent bonds with substantial deformation and Figs. 42(a) and (b) represent bonds with minimal deformation. The macrographs clearly show the difference.

5.4.5.2 OPTICAL MICROGRAPHY:

Fig 43(a) shows an optical micrograph at a low magnification of the area near the interface for the 25 Vol % SiCp/6061 Al MMC bonded sample shown in Figs. 38 and 41(a). The bonded sample was longitudinally cut, mounted, metallographically polished and etched as per procedure #1 of Section 4.4.7. Fig. 43(b) shows a close up view of the bond area, free of voids, at a higher magnification. Fig. 43(c) shows a close up view near the bond showing the formation of voids near the interface. Fig. 43(d) shows the optical microstructure in the base metal far away from the interface at the same magnification. Fig. 44 shows the optical microstructure of the sample seen in Fig. 41(b). It shows the bond area at low magnification of a 25 Vol % SiCp/6061 Al composite material liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer, with a 550 psi compressive loading at 5 milli-torr vacuum and a deformation of 35%.
Fig. 41 Macrographs of 25 Vol% SiCp/6061 Al MMC 3/8" diameter, liquid phase diffusion bonded with a 0.001" silver interlayer at 5 milli-torr vacuum; (a) at 570 °C for 10 min at 300 psi compression and a deformation of 27.6% and (b) at 580 °C for 20 min at 550 psi compression and a deformation of 35%.
Fig. 42 Macrographs of 25 Vol% SiC<sub>p</sub>/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer, at 580 °C for 20 minutes; (a) at 270 psi compression with 3% deformation and 400 milli-torr vacuum and (b) at 300 psi compression with 5% deformation and 5x10<sup>-5</sup> torr vacuum.
25 Vol% SiCp/6061 Al MMC 3/8" diameter, liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi, with a deformation at the interface of 27.6%; (a) Bond zone, note the flow lines disturbed near the interface (at A) and (b) a close up view suggesting particulate enrichment near the interface (in the area B - B).
Fig. 43 25 Vol% SiCp/6061 Al MMC 3/8" diameter, liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi, with a deformation at the interface of 27.6%, (c) a close up view of the bond zone showing voids (at arrows) and (d) base metal far away from the bond area (unetched).
Three zones similar to those witnessed in the previous sample are evident. Fig. 44 (b) reveals the curved flow lines in a close up view. Fig. 44 (c) shows voids near the interface and flow lines curving towards the interface. Figs. 45 are obtained from a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes with 270 psi compressive loading, a vacuum of 400 milli-torr and a deformation of 3% (sample shown in Fig. 42 (a)). In this case the thermocouple wires were percussion welded to the sample surface 2 mm from the interface. This sample was metallographically polished as per method #2 listed in the section 4.4.7 and was not etched. Figs. 45 (a) and (b) show a single void and a series of voids near the interface at a low magnification. Fig. 45 (c) shows an area largely free of voids at low magnification and Fig. 45 (d) shows an enlarged view of the central portion of the same area in Fig. 45 (a). The Figs. 45 also indicate that distinct zones occur near the interface. Another important observation of the optical micrography is the occurrence of voids near the interface.

5.4.5.2 SCANNING ELECTRON MICROSCOPY ANALYSIS

Fig. 7 showed an SEM micrograph of a 20 Vol% SiCp/6061 Al MMC material. From this picture, it can be observed that the extrusion process has a clear effect on the particulate morphology. Figs. 46 show SEM micrographs of the bond region of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001" silver interlayer at 575 °C for 30 minutes with 500 psi compression and a deformation of 7.6%. Fig. 46 (a) shows two zones near the bond interface. This figure clearly illustrates particulate enrichment near the interface.
Fig. 44 (a) 25 Vol% SiCp/6061 Al MMC 3/8" diameter, liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes at 5 milli-torr vacuum and a compressive load of 550 psi and a deformation of 35%, bond area at low magnification showing the three distinct zones.
25 Vol% SiCp/6061 Al MMC 3/8" diameter, liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes at 5 milli-torr vacuum and a compressive load of 550 psi and a deformation of 35%; (b) area adjacent to the bond interface and (c) Voids at the interface.
Fig. 45 25 Vol% SiC_p/6061 Al MMC 3/8" diameter, liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes at 400 milli-torr vacuum and a compressive load of 270 psi, with a deformation at the interface of 3%; at low magnification showing bond interface (a) with minimal voids and (b) highlighting a series of voids.
Fig. 45 25 Vol% SiC_p/6061 Al MMC 3/8 " diameter, liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 ° C for 20 minutes at 400 milli-torr vacuum and a compressive load of 270 psi, with a deformation at the interface of 3 %; bond zone (c) at low magnification and (d) showing minimal voids and particulate enrichment at the interface.
Fig. 46 (b), at a higher magnification, shows the morphology of particulate material and also the size of voids near the bond interface. Figs. 47 through 49 show the observations of interfacial voids. Figs. 47 (a) and (b) show the occurrence of voids in the interfacial region for a composite sample bonded at 580 °C for 20 minutes. The deformation is 1% computed as a change in total length to the original length of the sample. Fig. 47 (a) shows a few voids along the bond line. Fig. 47 (b) shows that the void region is associated with the particulates. It shows the different morphologies and the size distribution of the particulates. It also indicates the absence of any reinforcement-matrix interfacial debonding. Fig. 48 shows the bondline for another composite sample liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer with 7% deformation. Fig. 48 (a) shows interfacial voids at the bond line and Fig. 48 (b) shows the bond line at a higher magnification. Fig. 48 (c) shows an interfacial void at a high magnification. This figure shows the size distribution and the morphology of the particulates and also indicates an accumulation of the particulates near the bond interface. Figs. 49 show interfacial voids in the liquid phase diffusion bonded composite sample shown in Figs. 42 (a) and Fig. 45. Fig. 49 (a) shows an interfacial void at the bond line. Fig. 49 (b) shows the same at a high magnification. It can be clearly seen that there is no interfacial debonding between the matrix and the reinforcement. They also show the particulate morphology and size distribution. These micrographs were taken on an un-etched sample metallographically prepared as per method # 2 listed in Section 4.4.7. It may be noted that the void area is associated with particulates. Fig. 49 (c) shows, in the same sample, a void free area at the interface and Fig. 49 (d) shows a large interfacial void. The sharp corners at the ends of the void may be noted. Again Figs. 49 (c) and (d) show the particulate morphology, size distribution and confirm that there is no interfacial debonding between the matrix and the reinforcement.
Fig. 46 SEM micrograph of a 25 Vol% SiCp/6061 Al MMC 3/8" liquid phase diffusion bonded with a 0.001" silver interlayer at 575 °C for 30 minutes with 500 psi compression with a deformation of 7.6%; (a) shows particulate enrichment (at A) near the bond interface (shown by arrows) and (b) morphology and size distribution of particulates near the bond interface.
Fig. 47 25 Vol% SiC_p/6061 Al MMC 3\(\frac{1}{8}\)" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes with a linear deformation of 1%, (a) shows voids along the interface (shown by arrows) and (b) at 10000 X shows the particulates in the void. No debonding at the matrix-reinforcement interface may be observed.
Fig. 48 25 Vol% SiC_p/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 20 minutes with deformation at the interface of 7 %; (a) bond line shows voids and (b) shows particulates in and near the voids.
Fig. 48 (c)  25 Vol% SiCp/6061 Al MMC 3/8 " diameter bar liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 20 minutes with deformation at the interface of 7 %, at 10000 X. The location of a particulate in a void and the variation in the particulate size may be noted.
Fig. 49 25 Vol% SiC<sub>p</sub>/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 580°C for 20 minutes at 400 milli-torr vacuum and a compressive load of 270 psi, with a deformation at the interface of 3%; (a) shows a small void at the bond line (shown by arrows) and (b) at 15000 X shows the same. No particulate-matrix interfacial debonding may be noted.
25 Vol% SiC<sub>p</sub>/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes at 400 milli-torr vacuum and a compressive load of 270 psi, with a deformation at the interface of 3%; (c) shows a void free area at the bond line and (d) shows a large interfacial void. The sharp features of the void edge may be noted.
The scanning electron micrographs clearly show the results of base metal extrusion in terms of the morphology and size distribution of the particulates. They suggest that random and continuous interfacial voiding is also present. However, they indicate that no debonding occurs at the reinforcement-matrix interface.

5.4.6 CONFIRMATION OF THE DIFFUSIONAL PROCESSES:

Some of the important steps necessary to prove the occurrence of diffusional processes that ultimately result in the dissolution of the interlayer, formation of the eutectic liquid and consequent isothermal solidification are to show that: i) no original interlayer material remains at the interface; ii) a concentration gradient exists in the bonded sample, with the peak associated with the interface, from which point the concentration decreases into the base metal on either side of the interface and iii) the demonstration of lamellar (the morphology of the eutectic solidification product depends on the type of equilibrium phase diagram of the system) eutectic solidification product in the microstructure. The last item will certainly prove that the process is one of eutectic solidification and not entirely due to solid state bonding. The information that is presented here proves all the above points.

Optical and scanning electron micrographs did not reveal the occurrence of any original interlayer material or the formation of any intermetallics at the original bond interface. However, it is necessary to use microscopic chemical analysis of the interfacial region to confirm the same. Electron probe microanalysis (EPMA) with a CAMECA microprobe and energy-dispersive X-ray microanalysis (SEM-EDAX) on a JEOL scanning electron microscope fitted with the microanalysis equipment were used to
investigate the chemistry at and along a section transverse to the bonded interface of longitudinally sectioned, mounted and polished sections of the bonded samples.

Fig. 50 shows the results of chemical analysis of a 25 Vol % SiCp/6061 Al MMC sample liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 20 minutes conducted by the SEM-EDAX process. The figure shows the variation of aluminum, silicon and silver along a line normal to the bond interface. Fig. 51 shows the EPMA chemical analysis across the interface of a 25 Vol % SiCp/6061 Al MMC bonded with a 0.001 " silver interlayer at 570 °C for 10 minutes. At this point it may be pertinent to mention that the EPMA results were obtained with a probe size of 10 microns. This probe size, which is larger than the conventional probe of about 1.5 microns, was chosen to minimize the chances that the probe only samples the SiC particles. This is a special situation compared to the conventional ingot metallurgy alloys where the precipitates or dispersoids are much smaller in size than the SiC reinforcement of this study and consequently the regular probe size of 1.5 micron would suffice for their analysis. However, it was decided that the samples be further scrutinized with probes of smaller sizes than ten microns, to make sure that no data is overlooked due to the size of the probe already used. It was decided that probes of sizes 1.5 and 5 microns be used to verify that there is no probe size effect on the concentration profiles.

Figs. 51 through 53 show that the effects of the variation of the probe size on the chemical analysis. They represent the chemical analysis, by EPMA, of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 10 minutes at a deformation of 27.6 %. The analysis shown in these figures were obtained at an electron microprobe size of 10 microns, 5 microns and 1.5 micron
Fig. 50  SEM-EDAX analysis of the interfacial region of a 25 Vol % SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer, at 580 °C for 20 minutes showing the variation of aluminum, silicon and silver with distance from the interface.
Fig. 51 Chemical analysis of the region across the interface, by EPMA, of a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 ” silver interlayer, ten micron beam rastering.
Fig. 52 Chemical analysis of the region across the interface, by EPMA, of a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 " silver interlayer, five micron beam rastering.
Fig. 53 Chemical analysis of the region across the interface, by EPMA, of a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 " silver interlayer, one micron beam rastering.
respectively. Fig. 54 shows the chemistry profiles from EPMA of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer, with a ten micron probe size. Fig. 55 shows the aluminum and silver profiles of the same sample shown in the Figs. 51 through 53. The concentration gradients are easily observed. The concentration of silver was shown at a different scale than that of aluminum. The concentration gradients are easily observed. The concentration of silver was shown at a different scale than that of aluminum.

5.4.7 OCCURRENCE OF THE EUTECTIC LIQUID PHASE:

It has already been demonstrated in Section 5.4.6, through electron probe micro-analysis that no remnants of the original silver interlayer could be found at the bond interface. Also, it was observed that the maximum composition of silver at the interface was far less than the maximum solid solubility of silver in aluminum at the eutectic temperature. Obviously, it is not possible for the complete interlayer material to be diffused away in the solid state, due to the relatively short time of heating involved. The complete removal of the interlayer is only possible if the dissolution of the interlayer, extrusion of the liquid and complete isothermal solidification of the remaining liquid occur. It was decided that this fact could be verified by employing a thicker interlayer and shorter times at the bonding temperature whereby the eutectic liquid can be trapped resulting in the formation of the eutectic solidification products.
Fig. 54 Chemical analysis of the region across the interface, by EPMA, of a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer, ten micron beam rastering.
Fig. 55 Chemical analysis of the region across the interface, by EPMA, of a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 " silver interlayer, ten micron beam rastering, showing only aluminum and silver profiles (Note the difference in the scale for silver).
For the verification of the formation of eutectic liquid, a silver interlayer of 0.005 " thickness and a holding time of only five minutes at the bonding temperature were chosen. The isothermal solidification process is controlled by the movement of the solid-liquid interface, progressing from the solid into the liquid thereby progressively depleting the eutectic liquid. Since this is a diffusion controlled process, the distance the interface moves is proportional to the diffusion coefficient of the solute in the solid, the time at the temperature and a constant depending on the chemistry as given by the phase diagram at the bonding temperature. In section 2.3.8, this was shown to be given by:

\[
Y_3 = K_3 \left( 4 D_s t \right)^{1/2} \quad (9)
\]

where

- \( Y_3 \) = interface displacement,
- \( K_3 \) = a constant depending on the phase diagram data and
- \( D_s \) = the diffusion coefficient of the solute in the solid at the bonding temperature.

The above equation and equation (14) show that to isothermally solidify the eutectic liquid due to a 0.005 " interlayer, a solidification time of at least 25 times the isothermal solidification time of the 0.001 " thick interlayer is necessary. Hence, it is possible that with a bonding time of five minutes, eutectic solidification products can be observed near the interface, provided the bond withstands the solidification shrinkage and the thermal contraction stresses on the Gleeble. Accordingly an experiment was conducted where 25 Vol % SiCp/6061 Al samples were bonded with the 0.005 " silver interlayer at 579 °C for 4 minutes at a compressive load of 570 psi with no vacuum with the samples held in the stainless steel extension fixtures located in the hot jaws.
Figs. 56 (a) and (b) show the microstructures of the composite sample bonded with a 0.005 " silver interlayer as described before. Fig. 56 (a) shows the remnants of the interlayer at a low magnification and Fig. 56 (b) shows the same at a higher magnification. Both figures are from a sample bonded, longitudinally cut, mounted in epoxy and polished as per metallographic method # 2 listed in Section 4.4.7 and were in the un-etched condition.

Fig. 57 shows the silver trace superimposed on the sample photomicrograph, obtained on the electron microprobe for the same sample. Fig. 58 shows the silver trace, superimposed on a sample photomicrograph, obtained for a 25 Vol % SiCp/6061 Al MMC, liquid phase diffusion bonded at 570 ° C for 10 minutes with a 0.001 " silver interlayer.

Chemical analysis of the areas closer to the center of the interlayer were examined on the electron microprobe to obtain information regarding chemistry. Fig. 59 shows the variation of aluminum and silver concentration with reference to the center of the interlayer. Fig. 60 shows the over all chemistry in the same area.

5.4.8 MECHANICAL PROPERTIES:

The mechanical properties of some of the bonded samples were established by the micro-hardness evaluation, tensile testing and qualitative bend evaluation.
Fig. 56 Micrographs of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.005 " silver interlayer, at 579 °C for 4 minutes, in the un-etched condition; (a) at low magnification and (b) at high magnification. Note the lamellar eutectic solidification products and the remnant of the original interlayer at the center (shown at A).
Fig. 57  Silver trace superimposed on a photomicrograph (obtained by the EPMA) at low magnification of 25 Vol % SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.005 " silver interlayer, at 579 °C for 4 minutes, in the un-etched condition. The sharp silver peak near the remnants of the interlayer (shown by arrow) in the center of the micrograph may be noted.
Fig. 58 Silver trace superimposed on a photomicrograph, at low magnification (obtained by the EPMA) of 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001" silver interlayer, at 570 °C for 10 minutes, in the etched condition. The silver profile may be compared to the sharp peak witnessed in Fig 57. Microhardness indentations may be seen in the center of the micrograph.
Chemistry profiles of silver and aluminum, obtained on EPMA, with reference to the center of the foil shown in Figs. 56 and 57. Note that the center is almost pure silver whereas for a short distance from the center near eutectic composition is obtained in the area where lamellar eutectic solidification products are seen in Fig. 56.
Fig. 60 Chemistry of regions near the interface with reference to the center of the interlayer. Note the near total absence of Mg and Si in the area where eutectic solidification products were seen.
5.4.8.1 MICROHARDNESS EVALUATION:

Fig. 61 shows the evaluation of microhardness (on a line normal to the bond interface near the center of the bond), for a distance of 3.6 mm on either side of an "as bonded" interface for a 25 Vol % SiCp/6061 Al MMC sample liquid phase diffusion bonded at 570 °C for 10 minutes with a silver interlayer of 0.001 " thickness at a deformation of 27.6 % (the sample shown in Fig. 38). The figure shows a hardness increase in the immediate vicinity of the interface.

Fig. 62 shows the variation of microhardness (Knoop hardness obtained with a 100 gram load) in a liquid phase diffusion bonded 25 Vol % SiCp/6061 Al MMC. The specimen was bonded with a 0.001 " silver interlayer at 580 °C for 20 minutes at 460 psi compression and a deformation of 7.8 % and was held at 500 °C for 40 minutes at a compressive load of 1392 psi. A vacuum of 1 X 10^-4 torr was maintained during the bonding and holding cycles. The sample was heat treated after bonding to the schedule #1. The figure shows the hardness variation to a distance of 3.75 mm on either side of the bond interface. Similar to the previous case a high hardness at the interface may be noted. The hardness profiles in both Figs. 61 and 62 indicated similarities in sharp increase near the interface followed by a gradual decrease into the base material irrespective of the fact that the Fig. 61 and 62 represented as bonded and bonded and post-bond heat treated samples.
Fig. 61 Micro-hardness evaluation on a line normal to the interface (for 3.6 mm on either side of the interface) of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded at 570 °C for 10 minutes with a 0.001 " silver interlayer at 27.6 % deformation. (B. M.: Base material)
Fig. 62  Microhardness profile (for 3.75 mm on either side of the bond interface) for a 25 Vol % SiCp/6061 AL MMC liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer, and post bond heat treated as per schedule #1 (Deformation 7.8 %). B.M: Base metal; H. T: Heat treatment Schedule #1 (as per section 4.4.4.3).
Summarizing the hardness profiles, it is observed that the as bonded sample indicates a higher hardness than the base metal near the bond interface followed by a zone of marginally lower hardness than that of the base metal and a transition zone in between. In the case of bonded and heat treated sample, the zone of high hardness remains at the interface but the base metal hardness is retained for some distance from the high hardness zone. Thereafter, the hardness is marginally lower than that of the heat treated base metal. The hardness variations can be explained by a flow of particulate rich material towards the interface and a small amount of base metal property degradation which may not be corrected by post bond heat treatment. It may be recalled that the same trend was observed for the thermally simulated and heat treated samples as shown in the Fig. 27.

5.4.8.2 EVALUATION OF STRENGTH:

Results of the tension tests of the bonded samples is shown in Table. 4. The mechanical testing was conducted on post bond heat treated and machined samples. The samples were machined to ASTM E-8 and were tested at a displacement rate of 0.01 " per minute. It can be observed (within the experimental conditions followed in this study) that deformation, though necessary for the liquid phase diffusion bonding of aluminum base alloys, has no large effect on improving the bond strength of the aluminum based composite material used.

Qualitative bend testing indicated that the joint shown in Fig.38 (25 Vol% SiCp/6061 Al MMC Liquid phase bonded at 570 °C for 10 minutes with a 0.001" silver interlayer at 27.6 % deformation) has a lower bend ductility than the as received and not thermo-mechanically simulated base material.
Table 4  Mechanical testing results of the bonded joints.

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Bonding Conditions</th>
<th>Heat Treatment</th>
<th>Tensile Strength (psi)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>i) 579 C  ii) 500 C</td>
<td>530 C, 1 hr WQ 48 hr at RT</td>
<td>11087</td>
<td>0.575</td>
</tr>
<tr>
<td></td>
<td>i) 20  ii) 40</td>
<td>175 C, 2 hr AC</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>i) 579 C  ii) 500 C</td>
<td>530 C, 1 hr WQ 48 hr at RT</td>
<td>12895</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>i) 20  ii) 40</td>
<td>175 C, 2 hr AC</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>i) 575 C  ii) 500 C</td>
<td>530 C, 1 hr WQ 48 hr at RT</td>
<td>12110</td>
<td>0.22</td>
</tr>
<tr>
<td></td>
<td>i) 20  ii) 40</td>
<td>175 C, 3 hr AC</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

RT : Room temperature, WQ : Water quench, AC : Air cool.
5.4.9 **FRACTOGRAPHY:**

Fracture surfaces of the bend tested and tensile tested bonded samples were analyzed by scanning electron microscopy. To understand the fracture behavior in bending, a 25 Vol% SiCp/6061 Al MMC bar 3/8" in diameter (as received) was slit lengthwise and bent under three point bending set up. A two inch base metal sample (not exposed to any thermal cycling) was bent with the flat side up, against two 3/8" rollers, 35 mm (1.38") apart, with a 7/8" roller at the bottom. This was done at a displacement rate of 0.1" per minute. It was observed that the line of fracture was away from the transverse center line.

Fig. 63 shows an electron microscope view of the 25 Vol% SiCp/6061 Al MMC three point bend tested fracture surface. Ductile dimples may be noted from Fig. 63 (a) and (b) at low magnification. Fig. 63 (c) also shows a clear dimpled fracture (at a higher magnification) and absence of any matrix-reinforcement interfacial debonding. A similar bend test was conducted under identical conditions for a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded sample. This was the same sample shown in Fig. 38. The electron micrographs of the fracture surfaces are shown in Figs. 64. Figs. 64 (a) and (b) show the fracture surface at a low magnifications. Fig. 64 (c) shows the same fracture surface at a higher magnification. Fig. 65 (a) and (b) show the fracture surfaces of composite sample liquid phase diffusion bonded at 570 °C for 10 min with a 0.001" silver interlayer and a 1% linear deformation. The specimen was easily fractured. Figs. 65 show a dimple at a very high magnification.
Fig. 63 Fracture surface of a three point bend tested 25 Vol% SiCp/6061 Al MMC 3/8 " diameter (as received) bar cut lengthwise; (a) at low magnification shows dimples on fracture surface and (b) at a higher magnification shows particulates in addition to the dimples.
Fig. 63 (c) Fracture surface of a three point bend tested 25 Vol% SiCp/6061 Al MMC 3/8 " diameter bar cut lengthwise, view of dimpled fracture at high magnification. Note no interfacial debonding between the matrix and reinforcement.
Fig. 64 Fracture surface of a 25 Vol% SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi, with a deformation at the interface of 27.6 %, cut lengthwise and three point bend tested; (a) at low magnification and (b) at a higher magnification. Note the reduction dimple size as compared to Figs. 47.
Fig. 64 (c) Fracture surface of a 25 Vol % SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes at 5 milli-torr vacuum and a compressive load of 300 psi, with a deformation at the interface of 27.6%, cut lengthwise and three point bend tested; at a high magnification. Note the dimples and reduction size of dimples as compared to Figs. 48. No interfacial debonding between the matrix and the reinforcement is observed.
Fracture surface of a 25 Vol% SiC$_p$/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 570 °C for 10 minutes with a linear deformation at the interface of 1 %, (a) at 10000 X shows dimples and (b) at 20000 X shows an enlarged view of (a).
Figs. 66 show the fracture surfaces of a heat treated and tensile tested 25 Vol% SiCp/6061 Al MMC base material. Fig. 66 (a) shows the macroscopic fracture surface at a very low magnification and (b) and (c) show the matching halves of the fracture surface at a high magnification. Figs. 67 through 69 show the fracture surfaces of the tensile tested bonded samples. Fig. 67 (a) and (b) show the fracture surfaces of the sample in Table 4 with the highest tensile strength. Fig. 68 (a) shows a large void at the center of the sample. Fig. 68 (b) shows the same at a higher magnification. Fig. 68 (c) shows the fracture surface near the center at a high magnification. Figs. 69 show the fracture surface of the sample with a 12.1 ksi tensile strength as reported in Table 4. Fig. 69 (a) shows that the fracture is associated with both dimples and flat fracture features. Fig. 69 (b) shows cracking and particulate debonding at the matrix-particulate interface. Fig. 69 (c) and (d) show two halves of the fracture surface of the same sample. Fig. 69 (e) shows again dimples and voids and no debonds.

5.5.0 SUMMARY OF BOND DATA:

Table 5 provides a summary of typical bonds in the 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001" commercially pure silver interlayer. The table provides information on bonding at temperatures from 570 to 580 °C and for times ranging from 10 minutes to one hour with both HF/HNO₃ acid treatment and Methanol/HNO₃ electropolishing surface treatments.
Fig. 66  SEM fractographs of a 25 Vol% SiCp/6061 Al MMC base material; (a) macroscopic fracture pattern at a low magnification and (b) fracture surface at a high magnification shows dimples, voids and particulate fracture (at arrow). Note no interfacial debonding.
Fig. 66 (c) Fracture surface of a heat treated 25 Vol % SiCp/6061 Al MMC base material at a high magnification. Note the dimples, voids and particulate fracture (at arrows). Matching half of the fracture surface shown in Fig. 66 (b). No interfacial debonding between the matrix and the reinforcement is observed.
Fig. 67 Fracture surface (tensile tested) of a 25 Vol% SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes with a deformation at the interface of 8.1%; (a) at low magnification shows cracks and (b) at high magnification shows dimples and particulates. Note interfacial debonding at A.
Fracture surface of a 25 Vol% SiCp/6061 Al MMC 3/8" diameter bar liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes with a deformation at the interface of 8.1%; (a) shows void and (b) at high magnification shows an enlarged view of dimples and particulates. Note no interfacial debonding.
Fig. 68 (c) Fracture surface of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001" silver interlayer at 580 °C for 20 minutes with a deformation at the interface of 8.1%. The heavy particulate concentration may be noticed.
Fracture surface of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001" silver interlayer at 575 °C for 20 minutes with a deformation at the interface of 10%; (a) at 3000 X shows features of flat and dimpled fracture and (b) at higher magnification shows cracks and debonding.
Fig. 69 Fracture surface of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 20 minutes with a deformation at the interface of 10 %. (c) shows dimples, voids and particulate fracture and (d) at higher magnification, shows second half of fracture surface with dimples, voids and particulate breakage (at arrows).
Fig. 69(e)  Fracture surface of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 20 minutes with a deformation at the interface of 10 %. Fracture surface shows dimples and voids.
5.6.0 RESULTS FROM ADDITIONAL EXPERIMENTATION:

As Tables 4 and 5 show, the bonded samples exhibited lower tensile strength than that of the base material. The bonding temperature and time in the TLP process must be sufficient for the formation of eutectic liquid and the subsequent isothermal solidification. But, section 5.3.0 has shown that a degradation in the base material properties can be expected due to the exposure of the sample to an elevated temperature for prolonged periods of time. Hence, additional experiments were planned with reduced bonding time and temperature in order to minimize the MMC thermo-mechanical degradation and improve the bond mechanical properties whereby. These range of bonding process parameters (over those presented in Tables 4 and 5) are shown in Table 6. These specimen were machined from 0.55" diameter 25 Vol% SiCp/6061 Al MMC bar stock heat treated as per schedule #2 (section 4.4.4.3) and machined to design I (section 4.4.3). They were held directly in stainless steel fixtures after a surface treatment procedure consisting of polishing on 600 micron SiC paper and surface cleaning procedure II (section 4.4.6.1). The samples were bonded at 1 x 10^-4 torr vacuum with a 0.001" silver interlayer.

The following micrographs, both optical micrographs (of the polished longitudinal section) of the bonded sample and the SEM micrographs of the fracture surfaces of the failed specimen, help understand the lower strength levels attained in tension testing. Figs. 70 (a) to (d) show the optical macro and micrographs of the sample #5 in Table 6. This representatively shows the possible bond microstructures of the samples in Table 6. This 25 Vol % SiCp/6061 Al MMC (from the 0.55" diameter extrusion) was liquid phase diffusion bonded at 575 °C for 10 minutes with a 0.001"
Table 5: Summary of liquid phase diffusion bonds of 25 Vol % SiCp/6061 Al MMC with the 0.001" silver interlayer

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Surface Treatment</th>
<th>Bond Conditions</th>
<th>Heat Treatment</th>
<th>Tensile Strength psi</th>
<th>% Elongation</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>#1</td>
<td>10 Min 570 deg C 27.6 % None</td>
<td></td>
<td></td>
<td>M.S.</td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>#1</td>
<td>10 Min 570 deg C 41 % None</td>
<td></td>
<td></td>
<td>B. M.</td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>#1</td>
<td>20 Min 579 deg C 5.5 % Sch 1</td>
<td>11087</td>
<td>0.575</td>
<td>-----</td>
<td></td>
</tr>
<tr>
<td>4.</td>
<td>#1</td>
<td>20 Min 579 deg C 8.1 % Sch 1</td>
<td>12895</td>
<td>1.2</td>
<td>-----</td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>#1</td>
<td>20 Min 579 deg C 10 % Sch 2</td>
<td>12110</td>
<td>0.22</td>
<td>-----</td>
<td></td>
</tr>
<tr>
<td>6.</td>
<td>60 Min 580 deg C 4.4 % Sch 1</td>
<td></td>
<td></td>
<td>M.S / L.C.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.</td>
<td>#2/30Secs</td>
<td>30 Min 575 deg C 12.8 % Sch 2</td>
<td></td>
<td></td>
<td>B. M.</td>
<td></td>
</tr>
<tr>
<td>8.</td>
<td>#2/60Secs</td>
<td>30 Min 575 deg C 10.6 % Sch 2</td>
<td></td>
<td></td>
<td>B. M.</td>
<td></td>
</tr>
<tr>
<td>9.</td>
<td>#2/90Secs</td>
<td>30 Min 575 deg C 14.4 % Sch 2</td>
<td></td>
<td></td>
<td>B. M.</td>
<td></td>
</tr>
<tr>
<td>10.</td>
<td>#2/120Secs</td>
<td>30 Min 575 deg C 16.1 % Sch 2</td>
<td></td>
<td></td>
<td>B. M.</td>
<td></td>
</tr>
</tbody>
</table>

#1 and # 2 : Surface finish procedures II and III as per Section 4.4.6.1;
Sch 1 and 2 : Heat treatment schedules 1 and 2 as per Section 4.4.4.3;
M. S : Metallography Sample;
B. M : Broken in Machining;
L. C : Large Cracks noted.
Table 6  Results of Additional liquid phase diffusion bonds of 25 Vol % SiCp/6061 Al MMC with the 0.001'' silver interlayer

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Surface Treatment</th>
<th>Bond Conditions</th>
<th>loading</th>
<th>Tensile Strength</th>
<th>% Elongation</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Time Min</td>
<td>Temp degC</td>
<td>Deformn. %</td>
<td>psi</td>
<td>psi</td>
</tr>
<tr>
<td>1.</td>
<td># 1</td>
<td>5</td>
<td>570</td>
<td>20.9</td>
<td>170</td>
<td>1000</td>
</tr>
<tr>
<td>2.</td>
<td># 1</td>
<td>10</td>
<td>570</td>
<td>13.8</td>
<td>170</td>
<td>670</td>
</tr>
<tr>
<td>3.</td>
<td># 1</td>
<td>5</td>
<td>575</td>
<td>10.0</td>
<td>500</td>
<td>1515</td>
</tr>
<tr>
<td>4.</td>
<td># 1</td>
<td>10</td>
<td>575</td>
<td>12.0</td>
<td>170</td>
<td>1515</td>
</tr>
<tr>
<td>5.</td>
<td># 1</td>
<td>10</td>
<td>575</td>
<td>12.6</td>
<td>160</td>
<td>980</td>
</tr>
</tbody>
</table>

Surface treatment #1: 600 grit SiC polishing and surface finish procedure II as per Section 4.4.6.1;
Heat treatment: Schedule 1 as per Section 4.4.4.3;
Vacuum: 1 x 10^-4 torr;
Sample design: Design I as per section 4.4.3;
M. S.: Metallography Sample
Fig. 70 Optical macro and micrographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 10 minutes with a deformation at the interface of 12.6 %, (a) shows the bond region at a very low magnification. Note the misalignment between the bonded halves and (b) at higher magnification shows the large number of voids in the central region at the bond interface.
Optical micrographs of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 10 minutes with a deformation at the interface of 12.6 %; (c) the bond region near the edge at a high magnification (note voids (A) and particulate enrichment (B) at the interface); (d) at a very high magnification shows an enlarged view of the bond near the edge. Note the particulate free region in both figures and its curvature (C) in (d) suggesting material flow towards the interface.
silver interlayer at 12.6 % deformation. Fig. 70 (a) shows the macrograph at a low magnification. Fig. 70 (b) shows the optical micrograph at a low magnification of the center of the bond region. Fig. 70 (c) shows the micrograph of the upper portion of the bond region at an increased magnification. Fig. 70 (d) shows a very high magnification optical micrograph of the upper portion of the bond region. Figs. 71 (a) to (f) and 72 (a) to (d) show the SEM fractographs of the fracture surfaces of tensile specimen #1 and 3 in Table 6. Figs. 71 represents the fracture surface of a 25 Vol % SiCp/6061 Al MMC, liquid phase diffusion bonded at 570 °C for 5 minutes with a 0.001" silver interlayer with 20.9 % deformation (specimen #1 in Table 6). Figs. 71 (a) to (d) and (e) & (f) show fractographs of the matching halves of the fracture surfaces respectively. Figs. 71 (a) & (b) show low and high magnification views of the central region and (c) & (d) show low and high magnification views of the edge region of the fracture surface. The fractographs of the matching second half of the fracture surface shown in Figs. 71 (a) to (d) are shown in Figs. 71 (e) and (f).

Figs. 72 shows the fracture surface of a 25 Vol % SiCp/6061 Al MMC, liquid phase diffusion bonded at 575 °C for 5 minutes with a 0.001" silver interlayer with 10 % deformation (specimen #3 in Table 6). Fig. 72 (a) show low and high magnification views near the edge region of the fracture surface and. Figs. 72 (c) and (d) show the edge region at low and high magnification respectively.
Fig. 71 SEM fractographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 5 minutes with a deformation at the interface of 20.9 %; (a) shows a low magnification view of the central region of the fracture surface. Note voids; (b) a high magnification view of the above figure. Notice a large number of dimples, voids and a small number of particulates on the surface. Also note reinforcement - matrix interfacial debonding at A.
Fig. 71 SEM fractographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 5 minutes with a deformation at the interface of 20.9 %; (c) shows a low magnification view of the edge region of the fracture surface; (d) a high magnification view of the above figure. Notice an extremely small number of dimples (as compared to (b)), voids, some flat fracture features and a very small number of particulates on the surface.
Fig. 71 SEM fractographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 570 °C for 5 minutes with a deformation at the interface of 20.9 %, matching half of the surface shown in (a) to (d); (e) shows a low magnification view near the edge region of the fracture surface; (f) a high magnification view of the above figure. Notice a very limited number of dimples, flat fracture features (A) and voids on the surface.
Fig. 72 SEM fractographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001 " silver interlayer at 575 °C for 5 minutes with a deformation at the interface of 10% ; (a) shows a low magnification view near the edge region of the fracture surface; (b) a high magnification view of the above figure. Notice a very limited number of dimples, particulate fracture (at arrow), reinforcement - matrix interfacial debonding (A) and voids on the fracture surface.
Fig. 72 SEM fractographs of a 25 Vol% SiCp/6061 Al MMC liquid phase diffusion bonded with a 0.001" silver interlayer at 575 °C for 5 minutes with a deformation at the interface of 10%; (c) shows a low magnification view near the central region of the fracture surface; (d) a high magnification view of the above figure. Notice a very limited number of dimples, flat fracture features (A), particulate fracture (at arrow) and voids on the surface.
CHAPTER VI

6.0.0 DISCUSSION

6.1.0 BASE MATERIAL CHARACTERISTICS

The material used in this study behaves differently compared to the ingot metallurgy 6061Al alloy in view of the powder metallurgy route matrix material and the strengthening reinforcement material used. Hence it is necessary to understand the behavior of the composite material in terms of response to heat treatment and the phase transformation (solid-liquid) characteristics in comparison to the 6061 Al alloy. Reports from literature suggest artificial aging times from two hours to the longer times used for the conventional ingot metallurgy 6061Al alloys. The results of the optimization observed in this study are shown in Fig. 18. The shorter artificial aging time has arisen from the increased dislocation density and its effects on precipitation in the matrix alloy as suggested in the literature. Also, reports of similar dislocation interaction include that of a SiCp/8090 Al alloy as suggested by Hunt et.al. wherein increased dislocation density was one of the reasons for accelerated age hardening. Pan et.al. observed peak hardness at aging times of 6 hours in SiCp/2124 Al.
composites and a hardness profile similar to that seen in Fig. 18. Bhagat et al. reported that the presence of whiskers altered (accelerated) the precipitation hardening characteristics in a SiC<sub>W</sub>/6061 Al MMC. Based on the above results and discussion it may be concluded that: i) peak hardness is attained at earlier aging times in the case of 25 Vol% SiC<sub>p</sub>/6061 Al used in this study compared to the matrix alloy and ii) an aging time of 2 to 4 hours is adequate.

Differential scanning calorimetry taken up to verify the solidus temperature of the matrix alloy has shown some interesting observations. The heating trace shown in Fig. 19 suggested a deviation from linearity at about 585 °C, indicating a heat absorption. Fig. 20 showed two heat liberation steps at 625 and 550 °C during freezing. The first may be the indication of the 6061 alloy regular freezing process (a liquidus close to 625 °C) and the other showed a quick heat release at 550 °C. This second release strongly suggested that it was due to the ternary eutectic solidification in the Al-Si-Mg system at the temperature of 555 °C as given below:

\[
L \rightarrow Al + Mg_2Si + Si \quad (22)
\]

It is possible that some dissociation of Mg<sub>2</sub>Si might have occurred during the holding at 650 °C. The evaporative depletion of magnesium at the elevated temperature may have facilitated the possibility of providing silicon for the above reaction. In a study involving elevated temperature exposure of 15 Vol% alumina reinforced 6061Al alloy MMC, Sugunama et al. observed base metal strength degradation due to magnesium evaporation at temperatures greater than 580 °C (853 K). The dissociation of Mg<sub>2</sub>Si at elevated temperatures from 6061Al has also been suggested by Calderon et al. In
addition, some free silicon might have been liberated from the reaction between silicon carbide and the liquid aluminum as per equation (1) given in the section 2.3.1. Though it is conventional to utilize the cooling curve to make a prediction on the solidus point, in this case, due to the multiplicity of the reactions occurring the heating curve was used. On the basis of the on heating curve, it was observed that the solidus lies at around 585 °C. A similar result can also be obtained from the cooling curve. The point towards the end of primary solidification, where the cooling curve deviates from the curved cooling section to become a straight line, also indicates the same solidus temperature. From Fig. 21 it might be predicted that some formation of aluminum carbide has occurred due to reaction between SiC and molten aluminum as per equation (1) given in the section 2.3.1, which also supports the above argument. The trace shown in Fig. 22 was obtained to see if any transformations or reactions occurred in the base material due to the elevated temperatures and times employed in the bonding of the composite material. A time of 60 minutes at 580 °C provided the extreme conditions as regards the liquid phase bonding was concerned. If the DSC trace indicates no transformations or reactions due to the base metal heating alone, then it is unlikely that in the bonding process any reactions occur due to the base metal heating alone.

The deviation from linearity on heating evident in Fig. 23, occurred at a time of 8.55 minutes after the start indicating a solidus point of around 585 °C. In this case the heat absorption after reaching the melting point seemed to be minimal compared to the case of 20 minutes at 700 °C. This might be due to a very small extent of the formation of aluminum carbide. This point was amply supported by the cooling trace shown in the Fig. 24. The cooling trace was similar to that shown in Fig. 20 except that the secondary solidification seemed to be very small in extent compared to the primary solidification,
unlike that of the sample shown in Fig. 20. Fig. 24 also confirmed the solidus temperature of around 580 °C. In Fig. 25 an indication of the possible liquation of the secondary solidification product as noticed in Fig. 24 was observed.

The summary of the DSC studies indicated that there was no spectacular change in the solidus temperature of the 25 Vol% SiC\textsubscript{p}/6061 Al MMC from that of the 6061 Al alloy, which was anticipated. It also supported the observation of Devletian and others that elevated temperature interaction between liquid aluminum and silicon carbide results in the formation of aluminum carbide.

6.2.0 THERMAL AND THERMO-MECHANICAL SIMULATION

As has already been indicated earlier, the SiC\textsubscript{p}/6061 Al composite material used in this study differed from the ingot metallurgy 6061Al in more than one respect: its constituents i.e., (i) the ceramic reinforcement and its morphology; (ii) the matrix material and its processing (i.e., powder metallurgy route); (iii) the interface between the two, its extent and structure and (iv) the elevated temperature solid-liquid stage processing employed in the consolidation of the material. Thus, the prediction of the effects of thermal processing of SiC\textsubscript{p}/6061 Al MMC, solely based on the effects from the ingot metallurgy 6061Al would be highly inaccurate and misleading.

The results of the furnace simulation study were highly interesting. If we consider the information from the as thermally treated base material presented in Fig. 26, it could be observed that the thermal treatment temperature range might be divided into two regimes i.e., one at and below 550 °C and the other above it. At and below
550 °C, time and temperature cause a slight increase in hardness. The reason for this increase, though surprising, is not hard to explain. Earlier in the second chapter, the observation of Arsenault and Fisher on the generation of dislocations at the reinforcement - matrix interface, due to the differences in the coefficients of thermal expansion between the matrix and the reinforcement, had been reported. They indicated that the interfaces are a source of dislocation generation with the primary driving factor being the difference in the coefficients of thermal expansion between the matrix and the reinforcement. They annealed 20 Vol% b - SiC fiber reinforced 6061 Al and 20 Vol% a - SiC particulate reinforced 6061AI at 527 °C (800 K) and furnace cooled the specimens. From transmission electron micrographs, they observed that the dislocation density in the composites was \(2 \times 10^{10}/\text{cm}^2\) which was one to two orders of magnitude higher than that reported for a 50% hot rolled wrought alloy. They attributed the matrix strength to a high dislocation density and to the presence of very fine Al - Mg - Si GP zones. Flom and Arsenault also indicated that a good interfacial bond between aluminum and SiC is necessary for the generation of a high dislocation density. Kim and others measured dislocation density near the reinforcement - matrix interface in SiCp/2024 Al MMC's. They observed particle size dependence of dislocation density and significant dislocation generation (\(10^{10}/\text{cm}^2\)) at the interface. This was one order higher than that in the control alloy.

Since the MMC was in the as - fabricated condition before the thermal simulation, the predominant factor for the hardness increase seemed to be an increased dislocation generation and its influence. On the other hand, interface interactions due to the elevated temperature might result in the interfacial microstructural damage (to be explained later). Therefore, it can be postulated that there was a competition between the strengthening due
to the increased dislocation generation and the weakening due to the microstructural
damage. At temperatures at and below 550 °C, the former process wins the competition,
irrespective of the time at temperature.

However, as Fig. 26 indicates, when the temperature increases above 550 °C, both
temperature and time at temperature become significant factors. At temperatures of
570 and 580 °C (which are below the solidus temperature of the matrix alloy) there
does not appear to be a marked decrease in hardness with temperature and times past
twenty minutes. In the first twenty minutes the microstructural damage process seems to
override the dislocation strengthening. Thereafter, it does not seem to have a significant
effect.

The processes occurring around 560 and 580 °C can be understood if we recall
that, at these temperature a small amount of liquid may form due to the following invariant
reactions 114, 108:

\[
\begin{align*}
\text{Al} + \text{Si} & \rightarrow \text{L} \quad \text{at } 577 \degree \text{C} \\
\text{Al} + \text{FeSiAl}_5 + \text{Si} & \rightarrow \text{L} \quad \text{at } 575 \degree \text{C} \\
\text{Al} + \text{Si} + \text{FeMg}_3\text{Si}_6\text{Al}_2 & \rightarrow \text{L} \quad \text{at } 567 \degree \text{C} \\
\text{Al} + \text{Mg}_2\text{Si} + \text{Si} & \rightarrow \text{L} \quad \text{at } 555 \degree \text{C}
\end{align*}
\]

The liquid formation and the amount of liquid formed depend on the constituents
presence, their availability, the temperature and time for interaction. The decrease in
hardness witnessed at higher temperatures may be explained by this liquid formation and
additional factors. Significant reduction in hardness is also caused by the recovery and
recrystallization processes operating at the elevated temperatures. As the temperature is further increased to 590 °C, a steep decrease in hardness with temperature and time is witnessed. As this temperature is above the solidus temperature of the matrix alloy one can surmise that a partial liquid phase forms and damages the microstructure. Thus all the above factors contribute to microstructural softening which decisively overtakes the dislocation strengthening effect.

As evident from Fig. 27, in all the samples thermally simulated for five minutes and heat treated, there was a significant reduction in the hardness compared to the as received and heat treated base material (with no thermal simulation). This indicated that post thermal simulation heat treatment was unlikely to return the composite to the exact mechanical properties achievable in the non-thermal simulated and heat treated composite. The extent of difference (though small in the five minute sample) depended on time, temperature and loading at the temperature (as will be noted later). However, at treatment times exceeding five minutes, the results could be explained on a basis similar to that of the thermally simulated and non-heat treated material. The similarity was that the results could be grouped under two categories. But unlike those in the former case, the materials thermally simulated at 500, 550 and 570 °C formed one group and those treated at 580 and 590 °C formed another group. The results of the first group were understood invoking the same two mechanisms considered to be operating in the thermally simulated and non-heat treated material. In the five minute simulated sample microstructural damage appeared to have caused the reduction in the hardness whereas at longer times the dislocation strengthening seemed to have overtaken the former mechanism. In the case of the second group, there was no apparent increase in hardness after the initial decrease, with a further reduction with time in the 590 °C case. This may
be understood as arising from the irreversibility of microstructural damage due to the liquid phase formation and the other mechanisms such as recovery and recrystallization as outlined before. Fuller et al. compression tested 20 Vol% SiCp/6061 Al MMC (in the as cast condition) at temperatures up to 550 °C (1032 F) and at strain rates from $10^{-3}$ to 160 per sec. They concluded that dynamic recrystallization controls deformation at the higher temperatures and strain rates.

Thus the results of thermal simulation lead us to the conclusion that thermal treatment up to 570 °C may not cause a significant reduction in material properties, whereas the damage at a temperature of 580 °C depends on treatment time and at 590 °C temperature and time steeply increase the damage.

The base material hardness values in the as received condition and as received and heat treated condition may be taken as the values for the zero treatment time as shown in Figures 26 and 27. They are the Knoop hardness values (100 gram load) of 120 and 205 respectively. On comparing the results of Gleeble thermo-mechanical simulation as shown in Table 2 and Fig. 28 to the furnace simulation results shown in Fig. 27 (in both cases the samples were post-simulation heat treated), some observations may be made regarding the case of the five minute treatment time. A temperature of 550 °C in the case of Gleeble thermo-mechanical simulation and a temperature of 540 °C with the furnace simulation yielded almost identical micro-hardness values even though this value was lower (205 vs 183) than the as received and heat treated base material with no simulation. In other words, at 550 °C thermal excursion even for five minutes had the potential to soften the microstructure but deformation of the order used here had no apparent detrimental effect. Obviously the hardness improvement resulting from
increased dislocation density (resulting from the differences in the thermal expansion coefficient as stated earlier) was not compensated by the losses due to creep. However, as noted above, the composite hardness was lower than that in the non-simulated sample. Increasing the time at temperature from 5 to 30 minutes showed a small improvement in hardness at this temperature. On the other hand with further increase in time from 30 to 60 minutes the Gleeble samples showed a loss in microhardness value thus indicating that deformation at longer times effected the microstructure. At 570 °C, the hardness profile was similar to that of the 550 °C sample up to a simulation time of 30 minutes. Beyond that, times of 30 and 60 minutes showed differences in hardness between the two cases indicating that as temperature increases, the role played by deformation increases. At 580 °C a steep drop in the microhardness value from 216 to 152 was noted even for the five minute sample. In this case there was a difference between Gleeble thermo-mechanical simulation and the furnace simulation microhardness values (173 vs 152). This indicated that at temperatures close to the solidus both temperature and deformation have an effect on the microstructure (the discussion here pertains to deformations used in this study). In general, a treatment time of 60 minutes resulted in a decline in the hardness in all the cases with the steep decline (from 198 to 175) coming from the lowest treatment temperature i.e., 540 °C. However, the optical observations of the microstructure of the materials reported above in the Table 2, suggested that, too much reliance might not be placed on the hardness values in totality. As the Table 2 showed, pits, voids and cracks were noted in the optical microstructure.

In summation it may be surmised that thermal excursion for a time even as small as five minutes softens the microstructure in the 25 Vol% SiCp/6061Al MMC with the degree of softening increasing with the temperature of exposure. Further the furnace
simulation and the Gleeble thermo-mechanical simulation results may be broadly divided into three categories. Category I contains the results of samples 1 to 4 (540 °C and 5 minutes at 570 °C) where at a temperature of 550 °C time, temperature and deformation have minimal effects on the microstructure. With an increase in temperature to 570 °C both time at temperature and deformation become important. Category III includes the results of samples 6, 8 and 9 (60 minutes at 570 °C and 30 and 60 minutes at 580 °C) where longer times at 570 °C and intermediate and longer times at 580 °C with deformation, have an adverse effect on the microstructure. Finally the results of samples 5 and 7 (30 minutes at 570 and 5 minutes at 580 °C) with deformation appear to fall into a transition category II between the above two. Thus Category I includes cases where significant damage to the microstructure from time, temperature and deformation may not be expected other than that initially seen. All samples at 540 °C fall into this category. In category II temperature and deformation play an important role whereas for category III temperature, time and deformation play important roles. Most of the samples at 580 °C fall into this category. It may be concluded that the main mechanism acting for hardness improvement arises from the improved dislocation density whereas i) recovery ii) dynamic recrystallization iii) creep induced damage and iv) the formation of a small amount of liquid (as indicated earlier) contribute to the hardness decrease.

Table 3 showed the mechanical test results of Gleeble thermo-mechanical simulated samples. All Gleeble thermo-mechanical simulated samples shown in Tables 2 and 3 were heat treated after simulation to schedule #2 whereas the furnace simulated samples were heat treated to schedule #1 (as per section 4.4.4.3). As shown Fig. 18
and as already explained the only difference in these schedules was a change in aging time from 2 to 3 hours (Schedule #1 vs 2) and this has a small effect on the hardness values.

From the fracture surface microstructures of the Gleeble thermo-mechanically simulated (540 °C) sample shown in Fig. 29, a river-like pattern with stepped terraces and radial lines originating from a point somewhere near the outer edge of the surface were noted. The presence of steps, voids and dimples on the fracture surface were observed. In Fig. 29(c), the presence of dimples and particulate fracture were noted. Fig. 29(d) showed the dimples with the particulate reinforcement located in the dimples. Fig. 29 did not show any evidence of decohesion or voids at the reinforcement-matrix interface. It is possible that the fracture has occurred with microscopic ductility. From the above figures it may be concluded (considering the thermal simulation studies and the tensile properties reported) that thermo-mechanical treatment at 540 °C is not likely to degrade the base material properties. The samples thermo-mechanically treated at 565 °C for 30 minutes as shown in Figs. 30 represented a change in fracture morphology from those at 540 °C. Though the presence of numerous dimples were still noted, the fracture surface displayed an increased number of voids. In Fig. 30(a), stepped or terraced fracture features were noted. Fig. 30(b) showed the view near a crack opening. Here fibrous fracture features were observed. In a report on elevated temperature tensile testing of SiCp/6061 Al, Hunn identified drawing and smearing due to the extensive ductility in aluminum, at a temperature of 482 °C (900 F). Fig. 73 from Hunn, shows the same features of fibrous fracture as observed in the Fig. 30(b). However, in the case of Fig. 30(b) the testing was done at room temperature. The dimples observed in the Figures 30(c) and (d) were similar to those observed by Gungor et. al., in the elevated temperature (523 K) fracture surfaces of cast SiCp/
2014 Al MMC's. They observed "coarse voids and limited Al/SiC interfacial
decohesion" in the creep deformed specimen. However, in the sample shown in Fig. 30,
there was no indication of reinforcement - matrix interfacial decohesion. Based on the
similarity of the dimples and fibrous fracture features, it can be concluded that failure in
the above samples occurs with microscopic ductility. In the fracture surface features
witnessed so far, though voids have been noted, no reinforcement-matrix interfacial
decohesion was observed. In addition, the close similarity of features observed in Figs.
29 (d) and 30 (d) is very interesting. Voids, dimples and particulate material at the base
of the voids were noted in both the figures. There was again no interfacial debonding or
decohesion. These features suggested that the fracture in both case has proceeded partly
by micro-void coalescence and particulate fracture with microscopic ductility. The extent
of occurrence of either mode might be different in both these cases.

The fracture pattern of the sample thermo-mechanically treated at 575 °C for 30
minutes (Fig. 31 (a)) markedly differed from that of river-like and stepped fracture
features seen in Fig. 29 (a). Evidence of fibrous fracture and cracks were noted. The
size of voids in Fig. 31 (b) was larger than those in the 565 °C treated sample shown in
Fig. 30 (c). In addition, wavy features were also observed. In the Figs. 31 particulates
were not readily apparent on the fracture surface. It is possible that the failure might have
occurred largely through an increased elongation and failure of the matrix material, than
either through interfacial failure at the reinforcement - matrix interface or the fracture of
the particulates (clearly no interfacial debonding or decohesion is seen as was noted in the
creep deformation by Gungor 116 et.al.). Dimples and voids were noted in Fig. 32 (a)
pertaining to the 575 °C, 10 minute sample. The size of voids appeared to be smaller
than those presented in Fig. 31 (b) and comparable to those presented in Fig. 30 (c).
In Fig. 32 (b) voids, dimples and particulates were seen. Fig. 32 (c) clearly showed the formation of fibrous material and particulate fracture. Figs. 32 (c) and (d) indicated that some of the fibrous fracture features seen are associated with particulate material whereas the fibrous material seen in Figs. 31 (e), (f) and 32 (e) contains particulate material coated with matrix material similar to that witnessed by Birt and Johnson. Characterizing the fracture surfaces of tensile tested 30 Vol% SiCw/Al, they observed that whiskers were coated with a thin layer of matrix material. Arsenault and Pande fractured a SiCw/Al composite in a scanning auger microprobe. They interpreted a 100% Al signal from areas on the SiC whiskers as due to the aluminum diffusing into SiC. In addition, they obtained similar results with a STEM analysis. From the above information and from the fractographs in this study, it can be concluded that the particulate material in some cases is coated with the matrix material. The tensile strength (60.08 ksi) achieved by this sample also suggested that observations of particulate fracture rather than matrix yielding indicate the benefits associated with the composite materials for structural applications. The similarities observed in Figs. 29 (d), 30 (d) and 32 (f) which represent samples bonded at 540, 565 and 575 °C, suggested that the current thermo-mechanical treatment did not cause fractures that are completely lacking in ductility. The evidence also indicated that no reinforcement-matrix interfacial decohesion may be observed. Based on these studies it may be surmised that thermal treatments up to approximately 560 °C did not cause significant damage to the microstructure and properties. At temperatures above 560 °C some damage to the microstructure and some degradation in mechanical properties could be expected though the amount of degradation strongly depends on the time at the temperature. Finally, based on microhardness testing of simulated samples, significant degradation in mechanical properties might be anticipated at temperatures above 580 °C, with the degradation
Fig. 73 Fracture surface of SiCp/6061 Al tensile tested at 900 F (Ref. 115). Features of fibrous fracture similar to those observed in Fig. 30 (b) may be noticed.
steeply increasing with treatment time. However, in analyzing the results of the thermo-mechanical simulation as reported in the Table 3, it has to be borne in mind that only one sample was tested in each case due to material constraints.

6.3.0 SUMMARY OF THERMAL & THERMO-MECHANICAL SIMULATION:

The temperatures and times chosen for thermal simulation (both furnace and Gleeble) represented the broad range of temperatures and times to be expected in the liquid phase bonding process. Together with the Gleeble thermo-mechanical simulation they offered a possible way to identifying the response of the base metal to the liquid phase bonding thermo-mechanical cycle. The temperatures chosen (540 to 580 °C) represented the lower and upper limits considering the aluminum-silver eutectic system on one hand and the solidus temperature of the composite on the other hand. The deformation represented that likely possible in the bonding process.

The thermal and thermo-mechanical simulation studies provided interesting results. Furnace simulation studies broadly divided the temperatures into two regimes, for the times employed in this study. Temperatures up to 570 °C formed one group whereas 580 and 590 °C formed another group. Samples thermo-mechanically simulated on the Gleeble, displaying a similar tendency, divided the time-temperature regime into three parts. Finally, samples thermo-mechanically simulated and tension tested indicated a strong influence of time at temperature, at temperatures around 570 °C.

Fig. 74 summarizes the results of the Gleeble thermo-mechanical simulation. It may be observed that both the tensile strength and the microhardness (evaluated for a
Fig. 74 Results of Gleeble thermo-mechanical simulation for a simulation time of 30 minutes.
simulation time of 30 minutes) follow a similar trend. This can provide the base line comparison data for evaluating the bond strength. In addition the tensile strength at 580 °C can be obtained by extrapolation.

The variation of microhardness between the furnace and the Gleeble simulated samples was predictable. At comparable temperatures and times the furnace simulated samples had somewhat of a higher value. This was due to the fact that the former were subjected to thermal simulation only unlike the thermo-mechanical simulation undergone by the latter samples.

The fracture surfaces of tensile tested samples have not shown any reinforcement-matrix interfacial decohesion. Fracture features point to the feasibility of microscopic ductility. The elongation values of the above samples must be evaluated considering the limited ductility of the base material. Some fracture surface features point to the possibility of formation of voids and fibrous material (particularly in the 575 °C 30 minute sample) at the elevated temperature Gleeble thermo-mechanical simulation. Fig. 73 from Hunn (Ref. 115) supported the above observation. The possibility of association of the matrix material with the fibrous features was noted.

The conclusions that can be drawn based on the above studies are presented as listed below:

i) For the loading, temperatures and times employed in this study thermo-mechanical treatment of 25 Vol % SiCp/6061 Al MMC at temperatures above 500 °C results in base metal strength degradation.
The simulation results may be broadly categorized into three regimes. A temperature of 540 °C and short term exposures at elevated temperatures of 570 (less than 30 minutes) & 580 °C (around 10 minutes) do not significantly degrade the base metal properties. Longer times i.e., more than 30 minutes at 570 °C or more than around 10 minutes at 580 °C may lead to a large degradation in the base metal properties. Times of about 30 minutes at 570 °C or 10 minutes at 580 °C seem to be in the transitional regime.

These results support the earlier observations on the operating mechanisms for degradation in strength at the elevated temperatures in 25 Vol % SiCp/6061 Al material. Thus, it appears that the basic issues involved in the elevated temperature bonding of these materials are the strength degradation resulting from microstructural factors comprising recovery, recrystallization, creep deformation and a small amount of liquid formation and improvements from increased dislocation density (resulting from differences in thermal expansion coefficients of the reinforcement and the matrix alloy). A competition between these two mechanisms, based on the time & temperature and loading of application will decide the winner. Lower times and temperatures favor strength improvement from the dislocation density mechanism whereas at longer times and higher temperatures strength degradation from the microstructural damage mechanism prevails.

Fig. 75 provides a summary of the results of thermo - mechanical simulation. Fig. 76 illustrates the recommended bonding parameter zones for the liquid phase
diffusion bonding of 25 Vol % SiCp/6061 Al metal matrix composite material. The above figures have been constructed on the basis of the following information:

i) The eutectic temperature of the aluminum - silver binary system i. e. 567 °C;

ii) The solidus temperature of the composite material i. e. 582 °C;

iii) the furnace simulation studies;

iv) the conclusions drawn from the Gleeble thermo - mechanical simulation;

v) the results of the Gleeble thermo - mechanical simulation (for a simulation time of 30 minutes) as presented in Fig. 74 and

vi) the fracture surface analysis as presented in results and discussed above.

Fig. 75 is based on the fact that the simulation time, temperature regimes have been categorized in to three parts based on the furnace and Gleeble thermo - mechanical simulation studies. Fig. 76 provides a recommended bonding parameter zone considering:

i) the need for the bonding temperature to be a) above the aluminum - silver eutectic temperature for the liquid phase bonding process and b) below the solidus temperature of the matrix alloy for minimal degradation in properties and

ii) the need for the bonding time to be a) sufficient to allow isothermal solidification process to be completed and b) minimal to reduce the degradation in mechanical properties due to the elevated temperature exposure.
Fig. 75 Summary of the results of base metal thermo-mechanical simulation
Fig. 76 Summary of recommended bonding parameters zone based on the thermo-mechanical simulation studies.
6.4.0 Liquid Phase Diffusion Bonding:

The liquid phase bonding of the 6061 aluminum alloy with the Al-Si-Mg interlayer, which relied on the melting of a ternary eutectic interlayer at temperatures above 560 °C, demonstrated that the process could be used successfully for aluminum based alloys. At the same time it brought out several important problems such as: i) the need for a ductile interlayer so that it could provide exact surface matching at the interfaces; ii) the need for chemistry of the interlayer to match ternary eutectic composition so that either it is completely molten or which will completely liquate with the aid of diffusion from the base metal at the bonding temperature employed and iii) a proper thickness such that either the liquid can be completely solidified through isothermal solidification or some of the liquid can be extruded and the rest can be isothermally solidified. The Al-Si-Mg interlayer used was not ductile, did not have exact ternary eutectic chemistry (since the DTA result revealed that it will be completely molten at temperatures exceeding 596 °C) and that iii) even if it were to be completely molten at the bonding temperature, the bonding time would be much longer than would be feasible with the equipment employed due to its large thickness (0.004"). Hence further work was carried out with a silver interlayer.

6.4.1 Time for Isothermal Solidification of the Silver Interlayer:

In section 2.3.8.3 the time for isothermal solidification of 0.001" and 0.004" thick Al-Mg-Si interlayers were shown to be 1.5 and 24 hours respectively. In this section the time for isothermal solidification of a pure silver interlayer is estimated.
With the help of equation (11) the maximum width of the molten liquid after the dissolution of the interlayer due to diffusion and interaction with the base metal can be calculated:

\[
W_{\text{max}} = W_0 \left( 1 + \frac{C_{\text{L}} - C_{\text{La}}}{C_{\text{La}}} \frac{\rho_{\beta}}{\rho_{\alpha}} \right)
\]  

(11)

where \( W_{\text{max}} \) = final maximum width of molten zone,
\( W_0 \) = initial width of the interlayer = 0.001"
\( \rho_{\alpha} \) and \( \rho_{\beta} \) = densities of A and B,
\( C_{\text{L}} \) = composition of liquid in equilibrium with solid of composition \( C_{\text{La}} \), at the interface, at the temperature concerned,
\( C_{\text{L}} \) = composition of the bulk liquid.

In the aluminum-silver binary system the values of \( C_{\text{L}} \), \( C_{\text{La}} \) and \( C_{\text{L}} \) at 575 °C, as computed from the phase diagram 110 are 0.6825, 0.50 and 0.72 respectively. Literature values of the diffusion coefficient of silver in aluminum are as follows:

i) \( D_{\text{Ag/Al}} \) at 575 °C = 2.57\times10^{-9} \text{ cm}^2/\text{sec} \quad (\text{Ref. 117})

ii) \( D_{\text{Ag/Al}} \) at 575 °C = 3.96\times10^{-9} \text{ cm}^2/\text{sec} \quad (\text{Ref. 119})

iii) \( D_{\text{Ag/Al}} \) at 572 °C = 5.60\times10^{-9} \text{ cm}^2/\text{sec} \quad (\text{Ref. 120})

iv) \( D_{\text{Ag/Al}} \) at 575 °C = 8.8\times10^{-9} \text{ cm}^2/\text{sec} \quad (\text{Ref. 121})

These diffusion coefficients are chemical diffusion coefficients based on the systems Al-8.5 at% silver, Al-14 at% silver, Al-3.5 at% silver and Al-20 wt% silver respectively.
For the purposes of the system used in this study, the Al-14 at% silver composition is closest among those above. This value of diffusion coefficient was calculated based on the pre-exponential term ($D_0$) and the activation energy for diffusion ($Q$) obtained by Roberge et al. from aging studies at 175 °C. Accordingly, the value of $3.96 \times 10^{-9}$ cm$^2$/sec was chosen as the diffusion coefficient of silver in aluminum for the purposes of this study. Thus at 575 °C,

$$W_{\text{max}} = (0.00254) \{1 + [(0.72-0.6825) \times 10.5/0.50] \}.$$  

The value of $W_{\text{max}}$ was calculated to be 30.83 microns. Using equation (14) the time for isothermal solidification was calculated.

$$t_3 = \frac{W_{\text{max}}^2 C \alpha^2}{C_{\text{Cl}}^{\alpha}} \frac{\pi}{16D_s}$$  \hspace{1cm} (14)$$

where $D_s = $ Diffusion coefficient of the solute in the solid. Thus at 575 °C,

$$t_3 = (30.83 \times 10^{-4})^{2} \{[0.6825^{2} \times 3.1414] / [(0.72) \times 16 \times (3.96 \times 10^{-9})]\}.$$  

The time for isothermal solidification for an aluminum-silver foil of eutectic composition was calculated to be 15 minutes. This short solidification time is possible due to the extensive solid solubility of silver in aluminum i.e., 55.6 wt %, at the binary eutectic temperature 110. However, in the case of a commercially pure silver interlayer, additional time should be provided at the bonding temperature for the interface composition to equilibrate from unity to that of $C_{\text{Cl}}^{\alpha}$. In such a case the term $C_{\text{Cl}}$ in the equation (11) should be treated as equal to unity. Thus, the time for isothermal
solidification with a silver interlayer of commercial purity of 0.001 " thickness is 75 minutes. Times for isothermal solidification at 580 °C and 570 °C temperatures were also calculated based on the phase diagram data and the values of $D_0$ and $Q$ from Ref. 119. These were 86 minutes and 66 minutes respectively.

Thus, it could be seen that remarkable improvements in the process can be made by switching to a silver interlayer of 0.001 " thickness as compared to the Al-Si-Mg interlayer examined initially. As was obvious from the above analysis, the time for isothermal solidification could be further reduced if the initial thickness of the interlayer can be decreased. After checking with a few manufacturers, it was observed that 0.001 " is the smallest thickness of the silver interlayer commercially available. Hence it was decided that the same may be used and the excess liquid be extruded out.

6.4.2 LIQUID PHASE DIFFUSION BONDING OF THE COMPOSITE MATERIAL WITH THE SILVER INTERLAYER:

The results of liquid phase diffusion bonding of 6061 Al with the 0.001" silver interlayer, as shown in Figs. 36 and 37, demonstrated that the thermo-mechanical cycle caused cracking and formation of fibrous material even for short bonding times (5 to 10 minutes) at 570 °C temperature. Hence further bonds were made on the composite material.

The macrographs from longitudinally sectioned bonded composite material, as presented in Fig. 41 clearly showed the deformation at the interface. The optical microstructure shown in Fig. 43 provided interesting information. At a low magnification
Figs. 43 (a) and 44 (a) revealed the occurrence of distinct zones near the interface. The higher magnification micrograph in Fig. 43 (b) showed bonded areas free from voids. The micrographs revealed that neither any interlayer material remained nor any intermetallics formed at the interface. The optical microstructure as shown in the above figures indicated the occurrence of distinct zones near the interface. They were: i) a dark etching zone of about 80 micron width near the interface; ii) a diffuse flow zone of about 250 micron width on either side of the above zone where the base metal flow lines are disturbed and iii) the base metal beyond the above zone where the original extrusion flow lines were preserved. A higher magnification view of the area near the bond did not reveal that any of the base metal flow lines were curved from their normal pattern. However, a close examination of the second zone, in Fig. 44 (b) revealed that the flow lines in this zone curved upward indicating macro - deformation. From the Fig. 44 (c) it was observed that some particulate free bands existed in the microstructure. The occurrence of voids and presence of distinct zones at the interface was also shown by Figs. 45.

Thus far optical microstructure has demonstrated specific features such as occurrence of distinct zones, voids, flow lines and particulate free bands at the interface. It can be speculated from the observation of the distinct zones, that, particulate enrichment, possibly, occurs near the interface. This can be proved by: i) scanning electron microscopy; ii) chemical analysis (electron probe micro analysis) and iii) micro - hardness survey. The presence of voids can be understood to have arisen from differences in diffusivities of the different species (aluminum and silver). It is difficult to prove the occurrence or otherwise of the reinforcement - matrix interfacial decohesion.
solely on the basis of optical microscopy. This necessitates the use of scanning electron microscopy.

SEM analysis of the composite material bonds provided interesting information. The base material micrographs showed particulates of varying sizes and morphologies. This could be attributed to the extrusion process used in the primary processing of the composite material. Some of the distinguishing features of the microstructure observed by the SEM included the absence of interfacial debonding between the matrix and the reinforcement, presence of occasional and regular voids at the interface and the different sizes and morphologies of the particulate material. Fig. 46 clearly showed the specific features of the zones seen in optical microscopy. The purpose of SEM analysis of the bonded sample was to understand the effects of thermo-mechanical cycle on the reinforcement. Fig. 46 (a) proved that the zone closest to the bonded interface comprised of particulate enriched material. When Fig. 46 (b) was compared to Fig. 6 (a) (representing the base material) two aspects became very clear: i) the inter-particle spacing in Fig. 46 (b) was considerably smaller than in Fig. 6 (a). This proved that a higher concentration of particulates was present near the bond interface; ii) unlike the clearly evident extrusion-direction orientation of the particulates seen in Fig. 6 (a), the concentrated particulates in Fig. 46 (b) seemed to be random in orientation. Figs. 46 also showed that the interfacial voids were small in size (5 mm approx.) and that there was no reinforcement-matrix interfacial decohesion. Figs. 47 to 49 also showed the size of the voids. Fig. 47 clearly showed that some of the particulate material had an aspect ratio ranging up to 3 to 1, highlighting the fact that the extrusion process fractures the particulates. Figs. 47 (b) and 49 (b) proved this point. Smaller voids were evident in Figs. 47 & 49 (a) and (b) whereas a large void was seen in Fig. 49 (d).
Higher magnification figures in 46, 47 and 49 proved that no reinforcement - matrix interfacial decohesion existed after the bonding.

Thus SEM confirmed: i) the occurrence of definite zones near the interface; ii) particulate enrichment near the interface; iii) the formation of voids, both small and large near the interface; iv) absence of reinforcement - matrix interfacial decohesion and v) the presence of particulates of varying sizes, morphologies and aspect ratios.

The processes taken up to validate the eutectic solidification included chemical and microstructural analysis. Figs. 50 through 54 presented the results of the chemical analysis. From Fig. 50 it was observed that the maximum concentration of silver (at the interface) was far less than both the original concentration and the maximum solid solubility of silver in aluminum at the bonding temperature. In addition, concentration gradients existed for the elements aluminum and silicon near the interface. The concentration gradient in aluminum resulted from the interdiffusional processes occurring at the interface. The reason for the variation in silicon concentration will be discussed subsequently. Fig. 51 also confirmed the complete elimination of the silver interlayer from the interface by extrusion and diffusion. Also, the concentration gradients of silver increasing towards the interface and aluminum decreasing towards the interface were observed. Figs. 50 and 51 together confirmed that the interlayer was removed from the interface. It may also be noted that both these data values, obtained from different equipment, confirmed similar trends thus validating each other in the process.

The results of chemical analysis conducted with varying probe sizes (shown in Figs. 51 through 53) validated the use of the larger ten micron probe size for EPMA.
These results confirmed that the usage of a larger probe size had no adverse effect on identification of the chemistry of silver at the interface. The chemical analysis results obtained with the smallest achievable probe size, did not suggest the presence of remnants of any interlayer material or composition or intermetallic formation at the interface. On the contrary, smaller probe sizes were observed to cluster the aluminum and silicon results. This was the reason that the ten micron probe size was used, in the first place.

Fig. 54 had shown the chemistry profiles from EPMA of a 25 Vol % SiCp/6061 Al MMC liquid phase diffusion bonded at 580 °C for 20 minutes with a 0.001 " silver interlayer, with a ten micron probe size. A comparison of the Figs. 51 and 54 revealed that in the latter case the silver diffusion extended to a longer distance than in the former case. This was expected due to the larger temperature and the longer time employed.

Earlier in the section 5.4.6 it was mentioned that the steps necessary to prove the process of eutectic liquid formation and subsequent isothermal solidification included the verification of the absence of the original interlayer at the interface, existence of solute concentration gradients at the interface and the demonstration of lamellar eutectic solidification product at the interface. The chemical analyses, as mentioned before, have clearly shown that no silver interlayer remained nor that any intermetallic formed at the interface. The results also showed that concentration gradients of silver existed near the bond interface. Thus two of the three necessary requirements for confirming the occurrence of eutectic liquid formation and the isothermal solidification process have already been met. Hence, it was necessary to prove that the formation of eutectic liquid had occurred at the bonding temperature. If sufficient eutectic liquid is formed and time at the bonding temperature is considerably smaller than that is necessary for isothermal
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solidification, then it is possible to show eutectic solidification products at the interface.
If the dissolved interlayer is completely removed by extrusion and isothermal
solidification, then the maximum concentration of silver at the interface will correspond to
the maximum solid solubility of silver in aluminum at the eutectic temperature. At the
time of completion of holding at the bonding temperature, if tlie molten zone is too wide
to be cogppletely solidified isothermally, then afte-^W^w to room temperature, chemical
and microstructural analysis should reveal eutectic composition and eutectic solidification
products at the interface. If the time of holding at the bonding temperature is just
sufficient to completely solidify the liquid isothermally ( assuming very fast cooling
which does not allow any diffusion in the solid state during the cooling process ) chemical
analysis should reveal an interfacial composition corresponding to the maximum solid
solubility of silver in aluminum at the eutectic temperature. On the contrary, if the time at
the bonding temperature is sufficient to completely solidify the liquid isothermally, then
due to diffusion in the solid state after completion of isothermal solidification and during
cooling to room temperature, the interfacial composition ( as revealed by the chemical
analysis ) should be less than the maximum solid solubility of silver in aluminum at the
bonding temperature.

The information available from the Figs. 56 was very interesting. This
represented a sample bonded for a small time ( 4 minutes ) with a thick interlayer
( 0.005" ). Several features observed from these micrographs were that : i) clear
lamellar eutectic solidification products were seen in the remnants of the interlayer ; ii) it
appeared that part of the unfused ( to be confirmed by chemical analysis later ) silver
interlayer still remained in the center and iii) the eutectic solidification product seemed to
be too fragile to withstand the thermal contraction stresses and the polishing forces.


Thus, Figs. 56 (a) and (b) confirmed the morphology of the eutectic solidification product. Also, it was very clear from the micrographs that the silver peak was very sharp and distinct in Fig. 57 compared to that seen in Fig. 58. These figures also confirmed that the composite sample bonded with a 0.005" silver interlayer had much higher silver remaining at the interface, than the composite sample bonded with a 0.001" silver interlayer.

Figs. 59 and 60 confirmed that a small nearly pure region of silver remained at the center of the bond. The concentration of silver which was about 99% at this point, sharply dropped to about 78% within a short distance and remained steady for a short distance. This region was the same region showing the lamellar eutectic solidification product in the optical micrograph shown in the Fig. 56. The concentration decreased away after this region gradually returning to less than the maximum solid solubility of silver in aluminum at the eutectic temperature. A striking observation of Fig. 60 was the near total absence of silicon and magnesium in the region where the eutectic solidification product was witnessed.

Thus, combining the EPMA results of the bonded samples with 0.001" silver interlayer and 0.005" silver interlayer (which demonstrated absence of the original interlayer at the interface and the existence of solute concentration gradients at the interface) and the observation of lamellar eutectic solidification products of near eutectic composition in the bond area, it could be concluded that isothermal solidification of the eutectic liquid generated by the interaction of the interlayer with aluminum in the base metal occurred in the liquid phase bonding process.
By now it was clear that the eutectic solidification process occurred in the liquid phase diffusion bonding of 25 Vol% SiCp/6061 Al MMC with silver interlayer. It was also noted that the bonding process resulted in clear zones near the interface with the central zone being termed the particulate enriched zone. Though microstructural evidence in terms of micrographs was available to prove this, verification of the same by microhardness evaluation would confirm this.

It may be anticipated that the elevated temperature bonding might cause a decrease in hardness on a heat treated 6061 aluminum matrix, due to precipitate coarsening and dissolution. As seen in the Fig. 61, the hardness actually increased in the immediate vicinity of the interface, for the composite material. Within a short distance from the interface, it sharply dropped but was still higher than that of the extruded base material but lower than that of the heat treated base material. Fig. 61 showed that the hardness gradually returned to about 150 KHN about one mm from the bond interface. A small hardness increase was noted at about 2 mm from the bond interface. Beyond that it returned to about 150 KHN. The sample shown in Fig. 61 was surveyed as bonded and was not heat treated after bonding. The hardness increase at the bond interface appeared to have been caused by an increase in the particulate density at the bond interface. This was probably caused by the flow of particulate enriched material towards the bond interface during the deformation that occurred in the bonding process. It may be observed that the hardness gradually increases towards the bond interface. The high hardness associated with the bond interface prevailed only for a short distance on either side of the interface. This distance of about 80 microns compared favorably with the dark etching zone near the bond interface as revealed by optical micrography. Beyond this zone the hardness returned to about 150 KHN. This occurred in the second diffuse zone as
revealed by optical micrography. Beyond the second zone the hardness remained at this value but for a small increase at a distance of 2 mm from the interface. The peak hardness at the interface, in Fig. 62 is lower than that seen in Fig. 61. This was consistent with deformation levels in both the samples. The sample in Fig. 62 was bonded at a deformation of 7.8 % whereas the sample in Fig. 61 was bonded at a deformation of 27.6 %. This confirmed the earlier observation that the hardness increase at the interface might be due to the deformation occurring in the bonding process. In Fig. 62, the hardness pattern at other distances was different from that observed for the previous sample. Beyond the zone of high hardness, the hardness remained at the heat treated base metal value for about 650 microns from the bond interface. This was obviously the zone where heat treatment restored the base metal hardness. The hardness in the other areas is less ( about 180 KHN compared to 207 KHN ) than that for the base metal heat treated as per schedule # 1.

In understanding the hardness variations several important factors have to be taken into consideration: i) deformation levels were different in the samples; ii) bonding times and temperatures were different and iii) the sample in Fig. 62 was post-bond heat treated. In addition, the resistance heating process in the Gleeble would cause a temperature gradient in the sample with the peak temperature associated with the interface. Thus at different distances from the interface, different thermal conditions existed which along with the deformation processes due to the bonding were likely to cause the variation in the hardness. At the interface particulate enrichment overrode these effects. The least hardness observed in Fig. 62 (580 °C for 20 minutes, with 7.8% deformation) was consistent with the thermally simulated and post simulation heat-treated (170 KHN for 580 °C for 20 minutes treatment) and Gleeble thermo-mechanically simulated (165
KHN for 580 °C for 30 minutes at 10.7% deformation) samples reported earlier in sections 5.3.1 and 5.3.2 respectively. Thus microhardness observations corroborate the earlier observation of distinct zones at the interface.

Results of mechanical property evaluation by tensile testing established that the joints had lower mechanical properties than either the heat treated base material or thermo-mechanically simulated and post-simulation heat-treated base material. Thus, two facts stood out from these results: i) a certain minimum amount of deformation was necessary for the liquid phase bonding of the 25 Vol % SiCp/6061 aluminum MMC with a silver interlayer of 0.001 " thickness for the experimental conditions used in this study and ii) the joint strength was considerably lower than the base material. Earlier in section 2.3.7.2, it was shown that (from literature) the processes of contaminant removal and attainment of intimate contact at the interface through yield and creep deformation are necessary for solid state bonding. In addition, the results support Metzger's (Ref. 64) observation that for diffusion welding of aluminum alloys, a good amount of plastic deformation is required, even at very high temperatures, as reported earlier. Even in the eutectic diffusion bonding of aluminum composites (from the results of Enjo et al 61) it is obvious that a certain amount of deformation is usually associated with the bonding. For the liquid phase diffusion bonding of 25 Vol% SiCp/6061 Al MMC with a silver interlayer (in this study), it was observed that a minimum deformation of 6 to 10 % was necessary. This can be ascribed to the creep deformation occurring at the elevated bonding temperature employed.

The results of mechanical testing indicated that the joint efficiency ranged from 25.66 to 29.85 %. This efficiency was based on the joint strength of the bonded and heat
treated samples relative to the tensile strength obtained in the thermo-mechanically simulated (575 °C for 30 minutes at a deformation of 16.13 %) and heat treated base metal as shown in Table. 3. If the joint efficiency were to be compared based on a heat treated base material strength (not subject to thermo-mechanical simulation) it would work out to 16.3 to 18.9 %. However, these joint efficiencies are not realistic since in section 5.3 it had already been shown that the thermo-mechanical cycle used for bonding resulted in mechanical property degradation of the sample.

Fractographs of the base metal and the bonded sample bend and tensile fracture surfaces provided information on fracture characteristics of the bonded samples. It was observed that the dimples in the bend test fracture surfaces (Figs. 64) of the bonded samples were much smaller than those observed in the base metal bend test fracture surfaces as seen in Figs. 63. Dimples (in Fig. 64 (c)) smaller in size than those seen in Fig. 63 (c), were clearly observed on the fracture surface. Particulates could be observed at the base of the dimples. Particulate fracture was also noticed. No matrix-reinforcement interfacial debonding was seen. Figs. 64 suggested that the fracture in bending in the bonded composite, was not totally devoid of dimpled features and suggested that it was associated with lower ductility than in the case of the base material.

Fractographs of tension tested heat treated base material fracture surfaces revealed small and large dimples, voids and particulate fracture. No reinforcement-matrix interfacial decohesion was observed. The examination of fracture surfaces of the tensile tested joints revealed that some amount of microscopic ductility still prevailed in the composite bonds even though the specimen failed with inferior macroscopic ductility compared to the base metal. It has to be borne in mind that the ductility of the base
material is itself inferior to the ingot metallurgy 6061 Al matrix material. Cracks (Fig. 67 (a)) were seen on the fracture surface at low magnification and dimples, voids and a few particulates were observed on the fracture surface at high magnification (Fig. 67 (b) and 68 (b)). No interfacial debonding was observed in Fig 68 (b) whereas in Fig. 67 (b) interfacial debonding could be noted. Increased concentration of particulates (Fig. 68 (c)), responsible for the hardness increase as mentioned in the earlier section, was also observed. A few particulates have undergone debonding at the reinforcement-matrix interface. This suggested that near the interface there is a possibility for a small amount of particulate debonding in regions where heavy particulate concentration exists. The features of flat fracture were also noted (Fig. 69 (b)). Figs. 69 (c) and (d) gave the evidence that some particulate breakage has actually occurred and that the fracture was not totally devoid of particulate fracture. In addition dimples and voids were seen in both the figures. Limited debonding was noticed from these figures. The fracture surface examination also revealed that particulate breakage occurred thereby providing some strength. In summation, the bonded material fracture surface exhibited reduced ductility than the base material and the particulate breakage features indicated good base metal bond strength. Also from Table. 5 it was observed, that, electropolishing under the conditions used in this study, did not yield good bond strength as compared to the HF/HNO3 surface treatment.

Some interesting features of the bonds with a reduced bonding time (10 minutes at 575 °C) were revealed by Figs. 70. These figures have shown that some of the bonds were associated with misalignment (Fig. 70 (a)) and a large number of voids (Fig. 70 (b)) at the interface. Figs. 70 (c) and (d) confirmed particulate enrichment, material flow and the presence of particulate free bands in the microstructure. These
figures confirmed two important factors relevant to this study: i) misalignment between the parts to be bonded seriously affected the bond strength (since this results in loading the joint with a bending moment whereas the requirement in tension testing was to establish the uniaxial tensile strength); ii) the reduction in bonding time resulted in an increase in the number of voids at the interface. As shown in Table 6 the tensile strength achieved was quite poor as compared to the values shown in Table 5. An examination of fracture surfaces was expected to provide additional information in this regard.

Fractographs of the MMC sample bonded for 5 minutes at 570 °C with 20.9% deformation were provided in Figs. 71. Fig. 71 (b) has shown that the central region of the bond was associated with small dimples indicating microscopic ductility. Reinforcement - matrix interfacial decohesion was also noted. The fractographs of the edge region of the fracture surface (Figs. 71 (c) and (d)) of the same sample indicated that its features were different from that of the central region. Here, extremely small number, if any, of dimples were noted. In addition features of flat fracture and presence of a small number of particulates, on the fracture surface, were observed. Fractographs of the matching half of the fracture surface shown in the above figures (as shown in Figs. 71 (e) and (f)) confirmed the features of flat fracture and a small number of dimples.

Increasing the bonding temperature to 575 °C has caused a very small increase in tensile strength (sample #3 in Table 6). Consistent with this small increase particulate fracture was noticed in one case. To confirm that the tensile strength was poor compared to the samples in Table 4, this fracture surface has shown reinforcement - matrix interfacial decohesion (Fig. 72 (b)). The edge region of this fracture surface had a
small number of dimples compared to the central region (Fig. 72(d)). Fig. 72(d) has also shown features of flat fracture. Thus the samples shown in Table. 6 represented poor bonding conditions arising from misalignment of sample halves and an increase in the number of voids at the interface due to the small bonding time. This analysis had shown some interesting complexity in that longer bonding times (within the results of this experiment) show some improvement in strength due to minimization of interfacial voids and at the same time cause a reduction in strength due to thermo-mechanical degradation of the base material. Apparently, an appropriate bonding time has to be chosen with due consideration to these factors.

6.5.0 SUMMARY OF BONDING TRIALS:

The following material summarizes the bonding trials of liquid phase diffusion bonding in this study.

1) Effect of bonding parameters:

The bonding parameters studied in this experiment were: bonding temperature, time and deformation. From process considerations, the temperature is dictated by the requirement of the eutectic liquid generation. Time is dictated by the temperature and the thickness of the interlayer used since the amount of eutectic liquid generated depends on the initial interlayer thickness, other things being equal. As the studies of thermal and thermo-mechanical simulation indicated, time and temperature were inter-related due to the elevated temperatures necessary for bonding. Simulation indicated that a time of 30 minutes at 570°C may not be damage the base metal properties but at 580°C even a
time as short as 15 minutes becomes critical. Exposure beyond 30 minutes at 580 °C damages the base metal properties. Suganuma et al. observed that elevated temperature exposure degraded the strength of 15 Vol % alumina short fiber reinforced AA 6061 Al alloy. They indicated that the concentration of magnesium decreased with increasing temperature and time due to vaporization from the specimen surface and hence recommended shorter joining times. Thermal and thermo-mechanical simulation studies in the current study also indicated strength degradation for the composite. In this study, it was observed that with thermo-mechanical treatment beyond 575 °C for longer than 30 minutes a strength degradation was likely. Recovery and recrystallization (due to the elevated bonding temperature) further soften the microstructure. In addition, the possibility of formation of liquid from several eutectic liquation reactions leads to microstructural degradation and consequent strength reduction.

Figs. 77 and 78 show the macro and micrographs of samples bonded at 580 °C for 20 minutes and one hour respectively. The sample in Fig. 77 was additionally held at 500 °C for 40 minutes. The figures show that the sample in Fig. 77 had no cracking while the sample shown in Fig. 78 had an extensive cracking problem. This confirmed the temperature/time dependency.

Deformation beyond the minimum required, did not seem to have an appreciable effect on bond quality. In some cases, even with deformations higher than 10 % (the maximum reported for the data in Table.5) satisfactory bonds could not be obtained at 570, 575 or 580 °C (as shown in Table. 6). Table. 6 confirmed the same since the sample #1 in this table was bonded with a 20.9 % deformation and failed while loading into the machine.
Macrograph of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 20 minutes with a deformation at the interface of 7.8 %. The sample was additionally held at 500 °C for 40 minutes.
Macro and Micrographs of a 25 Vol% SiCp/6061 Al MMC, liquid phase diffusion bonded with a 0.001 " silver interlayer at 580 °C for 60 minutes with a deformation at the interface of 10%:
(a) macrograph showing extensive cracking transverse to the interface and (b) shows large voids on the bond line and a crack close to the bond line at a low magnification.
II) Surface finish effects:

The types of surfaces examined for this study included: i) as turned and chemically cleaned surfaces; ii) as turned & wire brushed and iii) as turned and electropolished surfaces. Surface finish has an important influence on the bond strength. Enjo et al., bonded 15 Vol % alumina short fiber reinforced 6063 aluminum and reported good strength values for the electropolished surfaces as compared to wire brushed and as turned surfaces. In the current study the electropolishing technique did not provide good bond strength. The electropolishing technique used in this study was different from that of the above referred authors. Lower polishing voltages and shorter bonding times were used. The joints held together very well on removal from the Gleeble and during heat treatment. But after heat treatment, they all failed during machining. The only differences was that they failed during different stages of machining.

III) Effects of interlayer thickness:

The interlayer thickness has a very important effect on the liquid phase bonding process due to its control over the time for isothermal solidification. In Section 2.3.8.1, it was shown that the time for isothermal solidification varied as the square of the interlayer thickness. The thermo-mechanical simulation studies proved that minimum time at the elevated temperature helps avoid base metal property degradation. Hence in order to have a good quality bond it is necessary to start with a smaller interlayer thickness. The minimum thickness of the silver interlayer commercially available for this study was 0.001 " (25.4 μm). Enjo et al. used a silver interlayer of 6μm (0.0025 " ) thickness in their diffusion bonding studies and used a bonding time of 30 minutes. This
is 4 times smaller than the thickness used in the current study. However, shear
deformation and eutectic liquid formation and extrusion likely reduced the bonding time in
this study.

IV) Joint mechanical properties:

In the results section it was shown that a joint tensile strength of 12.89 ksi
(88.98 Mpa) was obtained at a joint efficiency of about 30%. The joint efficiency was
based on the strength of thermo-mechanically simulated base metal. Enjo et al., in
their study on the joining of 15 Vol% alumina short fiber reinforced 6063 aluminum alloy
at 873 K (600 °C), reported joint tensile strength values of 145 and 188 Mpa (21 to 27
ksi) when the bonding was carried out with a silver interlayer of 6 mm (0.00025")
thickness and observed that the joint failed at the bond interface. Bond strength in the
current experiment may be further improved by obtaining a lower thickness interlayer and
carrying out the bonding for shorter times.

V) Microstructural characterization:

Optical micrography revealed the occurrence of distinct zones near the bond
interface. They included a particulate enriched zone at the bond interface and a diffuse
flow zone beyond the particulate enriched zone. Microhardness evaluation confirmed this
with a hardness elevation at the bond interface. The formation of these zones could have
been facilitated by the deformation occurring in the process. SEM analysis indicated
negligible particulate-matrix interfacial debonding. This removes any strength loss from
such debonding.
VI) **Fractography:**

Fractography demonstrated that some amount of microscopic ductility is retained in the bonded material (as seen from the dimples on the fracture surface). The macroscopic ductility is less than that of the as received base material. Limited particulate fracture, witnessed in the fracture surfaces of the bonded material, suggested good interfacial bond strength between the matrix & reinforcement and good strength for the joint itself. Lack of decohesion at the reinforcement - matrix interfaces (except in a few cases) also indicated that the thermo - mechanical processing in the bonding did not contribute to interfacial decohesion.

The reduction in the bond strength was attributed to i) property degradation due to the elevated temperature thermo - mechanical processing experienced in the bonding; ii) misalignment of the joint halves in the bonding process; iii) formation of interfacial voids and iv) particulate clustering at the reinforcement - matrix interface.
7.0.0 CONCLUSIONS

i) Thermal and thermo-mechanical simulation studies were conducted on a 25 Vol % SiCp/6061 Al to study the effects of temperature, time and loading on degradation of the composite material. On this basis, bonding conditions of temperatures from 567 to 580 °C and times up to 30 minutes were identified for subsequent liquid phase diffusion bonding studies with a silver interlayer.

ii) The 0.001" thick silver interlayer material was preferred to a ternary Al-Si-Mg interlayer to carry out the liquid phase bonding from ductility and bonding temperature considerations.

iii) Characterization of the bonded joint with optical microscopy confirmed the occurrence of distinct zones in the vicinity of the bond interface. These included a typically 80 micron width dark-etching particulate enriched
zone which contained the bond interface and showed a hardness higher than the matrix material. The next zone was identified as a diffuse flow zone of about 250 micron width showing a disturbed pattern of base material extrusion flow patterns. Beyond this, the microstructure typically represented that of an extruded base material.

iv) Based on the metallographic and micro-hardness observations it was concluded that the bonding thermo-mechanical conditions resulted in the flow of particulate enriched material towards the bond interface.

v) Optical microscopy of the bond with the 0.005" silver interlayer showed lamellar eutectic solidification products near the bond interface. EPMA of the above sample confirmed the composition of the lamellar solidification products to be of near eutectic composition. The above demonstrated the presence of eutectic liquid at the bonding temperature and the process of eutectic solidification in the liquid phase diffusion bonding technique.

vi) EPMA with varying probe sizes confirmed that no part of the original silver interlayer remained at the bond interface. It also showed that a silver gradient existed at the bond interface, with a peak at the bond interface and decreasing into the material on either side. The peak silver composition at the interface was observed to be less than the maximum solid solubility of silver in aluminum at the bonding temperature.
vii) The tensile strength of the bonded joint (579 °C for 20 minutes, at 12.9 ksi) was observed to be considerably less than that of the thermomechanically simulated (575 °C for 30 minutes) base material at 43.2 ksi resulting in a joint efficiency of 29.86%. However, compared to the strength of the non-simulated and heat treated material this joint efficiency is considerably lower. Decreases in joint strength can be attributed to voids at the interface.

viii) Based on the observations from optical micrography and SEM it can be concluded that voids occurred at the bond interface which contributed to the decrease in mechanical properties. The occurrence of the voids may have arisen from microscopic interfacial mismatch, Kirkendall porosity and entanglement of enriched particulates at the interface.

ix) Fractographic evidence pointed to the occurrence of limited microscopic ductility.

x) Time at temperature was identified to be very critical in view of the base metal property degradation likely to occur at the elevated temperatures employed for bonding. Also, it was observed that a minimum amount of deformation is necessary for the bonding of the material used in this study.
8.0.0 LIST OF REFERENCES


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