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Diffusion welding of an RS/PM aluminum alloy

Ananthanarayanan, V., Ph.D.
The Ohio State University, 1988
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UMI
DIFFUSION WELDING OF AN RS/PM ALUMINUM ALLOY

DISSERTATION

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

By

V. Ananthanarayanan, B.Tech., M.S.

The Ohio State University
1988

Dissertation Committee: C.E. Albright
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Approved by

Advisor

Department of Welding Engineering
To my dear wife Brindu
without whose understanding and cooperation
this venture would not have been possible
ACKNOWLEDGEMENTS

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1. INTRODUCTION

This introductory chapter briefly describes the development of advanced aluminum alloys for elevated-temperature structural applications. Heat-treatable ingot metallurgy (IM) alloys and sintered aluminum products (SAP) are known to exhibit limitations which have led to the development of rapidly solidified/powder metallurgy (RS/PM) alloys for elevated temperature service. However, the metastable microstructures and high gas contents of these RS/PM alloys restrict their possible joining methods. Solid-state joining methods, such as diffusion welding with silver interlayers may offer an effective joining method for these alloys.

This study investigated the diffusion welding of an RS/PM aluminum alloy with silver electroplated interlayers. The intention was to form a final joint consisting of ductile intermetallic compounds that were stable at the service temperature without excessive base material degradation during welding.
During the past two decades, benefits resulting from rapid solidification processing have been demonstrated for a number of metals and alloy systems. A wide variety of rapid solidification techniques providing particulate cooling rates from $10^2$ to $10^9^\circ$C/Sec (180 to $1.8 \times 10^9^\circ$F/Sec) have been utilized to generate a wide range of product forms, metallurgical structures and mechanical properties (Ref. 1). Significant metallurgical advantages offered by rapid solidification include: greatly extended solid solubility, minimization of alloying element segregation, refinement of grain size and the formation of unique metastable phases (Ref. 2).

The development of improved elevated-temperature aluminum alloys has been a subject of research and development for nearly a decade (Ref. 3). Aluminum alloys which exhibit higher strength and creep resistance offer the potential for lower weight and reduced costs in aerospace components through the replacement of heavier and more expensive materials like titanium. High strength ingot metallurgy aluminum alloys are limited to applications where temperatures do not exceed $176^\circ$C ($350^\circ$F), since precipitate coarsening and consequent loss of strength occur rapidly at higher temperatures.
Also, a stable microstructure with a high strength at elevated temperatures cannot be obtained exclusively by cold working the alloy or by solid-solution strengthening. However, producing a dispersion of an insoluble phase or phases in a face-centered cubic (alpha) aluminum matrix can effectively provide high strength coupled with microstructural stability at elevated temperatures (Ref. 4).

One approach used for the preparation of dispersion-hardened alloys was by pre-oxidation of aluminum powder or by blending aluminum powder with oxide powders (Ref. 5). The Sintered Aluminum Powder (SAP) alloys retain strengths up to the melting temperature. However, their useful ductility at high temperatures is impaired by pores opening at oxide-metal interfaces, and they also have limited room temperature strength.

The extended solid solubility resulting from rapid solidification permits the fabrication of alloys with a high volume fraction of fine intermetallic dispersoids, even when equilibrium solubility is low. If the elements forming these dispersoids have very low solubility and diffusivity (e.g., Fe, Ce and Mo in Al) in the alpha matrix in the solid state, they can act to stabilize
microstructures at elevated temperatures. The result can be the achievement of higher service temperatures not attainable with conventional ingot metallurgy alloys. Towner (Ref. 4) found that the ductility of atomized and extruded Al-8Fe powder alloys increased with increasing temperature in contrast to the SAP alloys, which decreased in ductility with increasing temperature.

As a result of Air Force supported research, a rapidly-solidified/powder metallurgy (RS/PM) Al-Fe-Ce alloy was developed at ALCOA Laboratories (Ref. 3). The alloy is a promising result of research and development work performed to identify alloying elements that have a high liquid solubility, a low solid solubility and low diffusion rates in aluminum. The attractive elevated-temperature strength and creep properties of the alloy have resulted in its continued development.

It is evident that a viable joining technique is an essential prerequisite to the efficient utilization of these materials for structural applications. Mechanical fastening using bolts, rivets, stitches, etc. results in areas of high stress concentration. This would result in a heavier design of the structure through the use of thicker materials. The crevices resulting from such
joining may also accentuate corrosion problems.

Much of the published research on Al-Fe-Ce alloys has concentrated on strength, corrosion, fatigue, magnetic and other properties with negligible results on joining. Two aspects should merit attention while considering a joining methodology for this class of alloys. The first is that the extremely fine microstructure of the alloy containing many metastable phases obtained through rapid solidification processing has to be "retained" or "recreated" in the weld fusion and heat-affected zones. This would be necessary for retaining the high temperature properties of the alloy. The second aspect is that the alloys inherently exhibit a high hydrogen content. This results from the formation of an oxide coating on the powder surfaces that tends to absorb moisture during storage and handling. Complete inert processing of the powder during the fabrication of the alloy may aid in minimizing the moisture content, but would increase the production cost. Degassing during the fabrication of the alloy is limited to temperatures that do not result in appreciable coarsening of dispersoids in the alloy and may not be effective in eliminating moisture/hydrogen released during the degassing process. The flue-gas atomized powder metallurgy Al-9Fe-4Ce alloy
studied in the present investigation had hydrogen contents about an order of magnitude higher than conventional ingot metallurgy (IM) aluminum alloys.

High energy density welding processes such as electron beam and laser welding may aid in suppressing the formation of coarse primary intermetallics and in "recreating" the metastable microstructures in the fusion zone of these alloys through rapid solidification of the weld metal. However, the high moisture and hydrogen contents of the Al-Fe-Ce alloy will likely result in problems due to porosity. Thus, possible joining methods for this alloy may be limited to solid-state processes such as diffusion welding and friction welding. The approach would be to "retain" the metastable microstructure of the base metal during joining and consequently its high temperature properties.

The purpose of this work was to study the diffusion welding characteristics of ALCOA's Al-9Fe-4Ce alloy. Of interest was the establishment of optimum time-temperature-pressure regimes for diffusion welding the RS/PM alloy using silver interlayers. In addition, metallurgical phenomena that limited bond-strengths in the RS/PM alloy were identified.
It was necessary to first establish a time-temperature-pressure regime for diffusion welding within which the alloy's microstructure would not coarsen significantly and thereby reduce strength. This involved a detailed study of the coarsening characteristics of the alloy under diffusion-welding thermo-mechanical conditions. Subsequently, cleaning procedures for the alloy required to produce surfaces of uniform activity for zincating and further silver plating were developed and optimized. Different parameters affecting the characteristics of the weld, including joint geometry and bonding pressure-time-temperature conditions were evaluated and optimized through destructive testing and in-depth microstructural characterization of diffusion welds.
2. BACKGROUND

In this chapter, the development and physical metallurgy of Al-Fe-Ce alloys is reviewed. Phases present in the extruded Al-Fe-Ce alloys are identified. Phase transformations occurring in the alloys upon exposure to different temperature-time regimes is reviewed. Fundamentals of deformation and diffusion welding including theories and mechanisms of the processes are discussed. The effect of diffusion welding process variables on joint properties is considered. The use of silver interlayers in the diffusion welding of aluminum alloys and their methods of deposition are explained.

2.1 Physical Metallurgy of Al-Fe and Al-Fe-Ce alloys.

Aluminum and iron (Ref.6) form a eutectic between the face-centered cubic alpha phase (solid-solution of Fe in Al with very little solid-solubility) and Al Fe at a composition of 1.7 wt % Fe and a temperature of 655 °C (1211 °F), as seen from the phase diagram in Figure 1.
Iron content, weight %

Figure 1. Al-Fe phase diagram with the superimposed coupled-eutectic (hatched) region (Refs. 8 & 9).
Soheil and Masuda (Ref. 7) quenched Al-Fe alloy drops in liquid sodium at two temperatures below the eutectic temperature. They observed fully eutectic structures over narrow composition and quenching temperature ranges even for hypereutectic compositions. The composition ranges over which eutectic structures were retained shifted with decreasing bath temperatures (increased undercooling) from the equilibrium 1.7 wt % Fe to as high as 6 wt % Fe. These regions were delineated as a coupled region on a plot of bath temperature versus composition and are superimposed on the Al-Fe phase diagram shown in Figure 1 (Ref. 8). At sufficiently high undercooling, a hypereutectic composition may solidify hypoeutectically, if its composition and the temperature to which it has been undercooled fall to the left of the coupled region.

Jones (Ref. 9) observed structural transitions in aluminum alloys hardened by rapid solidification. The alloys were with Fe and other first series transition elements and were rapidly solidified by splat-cooling from the melt. Al-Fe alloys showed a hardened surface layer of limited etching response, which he termed zone A, and a dark-etching, softer central layer, which he termed Zone B. The extent of Zone A from the surface inwards increased with decreasing Fe content and increasing cooling rate.
during splat-cooling. Microanalysis showed no significant differences in iron content between or within the zones. Zone A had at least twice the hardness of Zone B, which, in turn, had about twice the hardness of a chill-cast alloy of the same composition. X-ray diffraction of Zone A regions showed broadening and shifting of alpha-Al reflections implying supersaturation with solute. No second phase reflections were detected. Alpha-Al reflections from Zone B regions were not broadened or shifted from standard reflections. Second phase reflections were present from FeAl₆ in Zone B regions of alloys containing 4-20% Fe. This is a metastable phase not seen in the equilibrium phase diagram. Selected-area diffraction patterns and dark field electron microscopy showed Zone A to consist predominantly of cellular or "dendritic-like" alpha-Al solid solution with a common orientation within each dendrite colony, as in corresponding cast or weld structures. The interdendritic network phases could not be conclusively identified. Primary phase particles in Zone B regions were also confirmed to be predominantly FeAl. Zone B regions had a coarser network structure and intermetallics were spaced a micrometer apart.
Figure 2 shows a 500 °C (932 °F) isotherm of the Al rich end of the Al-Fe-Ce phase diagram reported by Zarechnyuch, Mis'kiv and Ryabov (Ref. 10). The binary phases in equilibrium with Al are Al$_3$Fe and Al$_4$Ce and the only ternary phase in equilibrium with Al is seen to be Al$_{10}$Fe$_2$Ce.

Angers (Ref. 11) studied particle coarsening in Al-Fe-Ce alloys at temperatures ranging from 316 °C (600 °F) to 575 °C (1067 °F) for times from 24 to 2160 hours. Specimens heat treated at temperatures of 475 and 575 °C (887 and 1067 °F) for two hours were also studied. In the as-extruded Al-8.8Fe-3.7Ce alloy, five dispersed phases were observed: metastable Al$_6$Fe, two metastable Al-Fe-Ce phases and equilibrium Al$_3$Fe and Al$_{10}$Fe$_2$Ce. One metastable Al-Fe-Ce phase had an Fe/Ce atom % ratio of 1 and the other Al-Fe-Ce phase had an Fe/Ce ratio of 4. Regions which appeared to be "decomposed Zone A" were observed in the as-extruded alloy. These regions were several microns in diameter and had solidified with a Zone A microstructure. Subsequent processing of the powder by hot pressing and extrusion had "decomposed" the microstructure in these regions. Electron-diffraction showed these areas to contain Al$_3$Fe, an equilibrium precipitate. However, STEM analysis showed that these
Figure 2. A 500 °C (932 °F) isotherm of the Al-rich end of the Al-Fe-Ce phase diagram (Ref. 10).
precipitates contained significant quantities of Ce. A large fraction of dispersoids in heat treated samples were equilibrium phases. In samples heat treated at 316 °C (600 °F) for long times, no Al₆Fe was detected and rarely were Al-Fe-Ce particles with Fe/Ce ratio of 1 detected. Reduced quantities of the Al-Fe-Ce phase Fe/Ce ratio of 4 were found. A large portion of dispersoids in the samples heat treated at 316 °C (600 °F) were of equilibrium nature. No non-equilibrium phases were found in samples heat treated at 475 and 575 °C (887 and 1067 °F) for two hours. Creep deformation of the alloy was found to enhance the rate of particle growth. In summary, heat treatment of the alloy tends to result in the formation of equilibrium Al₃Fe and Al₁₀Fe₂Ce at the expense of metastable phases. The metastable phase Al₆Fe appears to be the fastest one to transform to an equilibrium phase upon heat treatment and the ternary Al-Fe-Ce metastable phases transform more slowly. Among the two Al-Fe-Ce ternary phases, the phase with an Fe/Ce ratio of 1 transformed more rapidly to an equilibrium phase/phases than the ternary phase with an Fe/Ce ratio of 4.

Figure 3 (Ref. 12) compares the elevated-temperature strength of an RS/PM Al-Fe-Ce alloy with that of ingot
metallurgy (IM) alloys 2024-T351 and 2219-T851. The Al-
Fe-Ce alloy was tested after soaking for 1000 hours at
150 and 232 °C (300 and 450 °F) and 100 hours at 315 °C
(600 °F). The RS/PM alloy is seen to be vastly superior
to the IM alloys for elevated-temperature service.

2.2 Diffusion welding
Diffusion welding is defined as a solid-state welding
process which produces coalescence of the faying surfaces
by the application of pressure at elevated temperatures.
The process ideally does not involve macroscopic
deformation, melting or relative motion of parts. A solid
filler metal interlayer (diffusion aid) may or may not be
placed between the faying surfaces (Ref. 13). The
principal mechanism for joint formation is solid-state
diffusion.

The expense and geometric limitations preclude
diffusion welding from replacing fusion welding on a
large scale. However, the higher cost may be tolerable in
many engineering situations where melting may not be
tolerated, high temperatures may have to be avoided or
materials may otherwise be impossible to join by fusion
welding (Ref. 14). Additional advantages of diffusion
welding include the minimization of distortion and
Figure 3. Tensile strength versus test temperature for RS/PM Al-8Fe-7Ce alloy and IM 2024-T351 and 2219-T851 alloys. The Al-Fe-Ce alloy was tested after soaking for 1000 hours at 150 and 232 °C (300 and 450 °F) and 100 hours at 315 °C (600 °F) (Ref. 12).
residual stresses, reduced requirements for post-weld machining, and the ability to bond large and inaccessible areas.

Solid-state welding can also facilitate the joining of dissimilar metals which may form brittle intermetallic compounds during fusion welding. The rate of formation of these compounds during solid-state welding would be lower than during fusion welding. Also, suitable interlayers may be used to minimize the formation of brittle intermetallic compounds.

Finally, solid-state welding is useful when high temperatures may degrade the properties of the base material. They may allow joining of materials strengthened by metastable phases that may be destroyed by fusion welding, such as SAP alloys and rapidly-solidified materials.

2.3 Fundamentals of solid-state welding

Atoms tend to bond to each other if they are brought closer to each other than $10 \times 10^{-10}$ meter and particularly within interatomic distances of $1-5 \times 10^{-10}$ meter. In practical applications, surface irregularities, metal oxides and contaminants act as
barriers to welding. Table 1 shows typical sizes of surface asperities in cut, machined or polished surfaces (Ref. 15). It is shown that even finely polished specimens exhibit asperities orders of magnitude larger than interatomic distances.

In addition to asperities, a metallic surface may exhibit an oxidized or otherwise chemically reacted surface and adsorbed gas, moisture or both. Figure 4 shows a schematic representation of typical metallic surfaces that may be brought into contact for solid-state joining (Ref. 16). The diffusion welding process should be capable of disrupting and dispersing surface contaminants thereby promoting mechanical intimacy of contact of the faying surfaces within interatomic distances. Though oxides tend to adhere strongly to metals and may form strong bonds with oxides, they are usually hard and brittle. Thus, the elastic strain energy stored in flattening the surface asperities may be sufficient to break the weld formed between brittle oxide layers once the welding pressure is released.

Oxides can be mechanically dispersed by deforming the metals during welding or by raising the temperature sufficiently to promote oxide dissolution. Deformation
Table 1. Typical sizes of surface asperities (Ref. 15)

<table>
<thead>
<tr>
<th>Process</th>
<th>Size of surface asperities (micrometers)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saw cutting</td>
<td>1.6 to 50</td>
</tr>
<tr>
<td>Normal finish machining</td>
<td>0.8 to 3.2</td>
</tr>
<tr>
<td>Fine polishing</td>
<td>0.05 to 0.40</td>
</tr>
</tbody>
</table>

Figure 4. Schematic representation of metallic surfaces showing surface asperities and oxide/adsorbed layers (Ref. 16). Hatched areas represent bulk metal, gray areas surface layers and the unmarked area the gap between the two surfaces.
welding relies on gross plastic deformation to disrupt the oxide layer and achieve contact between large areas of fresh metal surfaces. This process is difficult to use at lower temperatures owing to higher flow stresses needed and is normally not applicable to a finished structural material.

Diffusion welding is a joining process which ideally involves no macroscopic deformation. Achieving extensive metallic bonding involves several steps. A model proposed for diffusion welding includes the following steps: initial surface contact along the asperities, deformation of surface asperities and interfacial boundary formation and elimination of pores through grain boundary migration and volume diffusion (Ref.13).

In the following sections, some basic aspects of deformation bonding, mechanisms of diffusion welding, and the effect of different welding variables on the properties of diffusion-welded joints are reviewed.
2.4 Deformation Bonding

Milner and Rowe (Ref. 17) review in detail the fundamentals of solid-state welding. They list indentation welding, upset butt welding and roll bonding as the then well established solid-state welding processes involving deformation at the faying surfaces. They define deformation as \((A_f - A_i / A_i)\), where \(A_i\) is the original area and \(A_f\) is the final area. For any two pieces of metal to be welded, a critical threshold deformation below which no welding occurred was found to exist. At room temperature, aluminum was found to require a 40% deformation to initiate welding, while lead and tin needed 10-15% and iron and nickel 60% deformation. They found a general trend towards higher deformations being required with increasing melting point or hardness of the metal. The threshold deformation was found to decrease with increasing welding temperature. Strong welds could be obtained at much lower deformations if there was relative movement of the faying surfaces while welding.

Nicholas and Milner (Ref. 18) investigated the roll-bonding of commercial purity aluminum at room temperature.
They found that the threshold deformation was 40% and that the joint strength increased with deformation above 40%. The joint strength did not become equal to the parent metal strength until about 60-70% deformation was attained. Trichloro-ethylene used as a solvent cleaner prior to bonding acted as a contaminant and was found to be a barrier to the achievement of complete bonding. Contamination by water vapor was not found to affect bonding above the threshold deformation.

Hauser, Kammer and Dedrick (Ref. 19) investigated the solid-state welding of aluminum alloys 1100 and 1170 of various tempers. The role of deformation and plastic flow was to break up surface films and bring clean surfaces into contact. At a joining temperature of 482 °C (900 °F), weld strengths greater than that of the parent metal strengths were obtained with 10% deformation. The threshold deformation decreased linearly with increasing temperature in the range of 204-554 °C (400-1000 °F). At a joining temperature of 482 °C (900 °F), joining time was found to have only a minor influence on weld strength if the threshold deformation was exceeded. The non-uniformity of interfacial extension across the faying surface was measured from a grid placed between the sheets. For a deformation of 50%, the interfacial
extension at the center of the strip could exceed 240%, while the sheet edge experienced no interfacial extension.

Albright (Ref. 20) examined the cause of embrittlement in steel-aluminum deformation welds. A pure aluminum cylinder was compressed between two ground and polished steel surfaces. Since an earlier study had shown that interdiffusion is greatly suppressed by high concentrations of oxygen in the steel, a highly deoxidized steel (0.0008% by weight oxygen) was used in the study. Welding was performed by forging the aluminum to a very thin sheet between the steel surfaces. The high pressures developed in the much softer aluminum due to elastic constraint were found to promote welding. Large interfacial shear developed between the aluminum and the steel was also found to promote welding. The thin aluminum sheet experienced an increase in surface area several times its original area, cracking the original oxide and exposing virgin metal. Welded specimens were heat treated at temperatures ranging from 360 to 440°C (680-824°F) for times up to 32 hours. A variety of evaluation techniques including metallography, microprobe and x-ray analysis, fractography and fracture mechanics tests were used to study the growth and morphology of
intermetallic compounds and voids and their effect on fracture toughness. The steel side of the fracture surfaces was decorated with dome shaped particles of Fe$_2$Al$_5$. With increasing heating time, Kirkendall porosity at the aluminum-Fe$_2$Al$_5$ interface was found to embrittle the weld rather than the intermetallic compounds. The porosity could cause a complete separation between the aluminum and the intermetallic layer, thereby stopping its growth altogether.

McEwan and Milner (Ref. 21) studied the roll bonding of dissimilar metals and classified them into four groups: immiscible, partially-miscible, miscible and intermetallic forming. Insoluble pairs of metals were found to weld readily. Systems possessing a high degree of miscibility welded readily, but were subject to diffusional porosity during high temperature service. Threshold deformation for aluminum was found to be 40% and the joint approached parent metal strength at deformations near 70%. Once the threshold deformation was well exceeded, experimental bond strengths were found to agree with theoretically calculated values based on oxide fragmentation for copper, aluminum, tin and lead.

Cline (Ref. 22) studied the deformation and diffusion
welding of 6061 aluminum alloy and a Ti-6Al-4V alloy. He discussed two theories on the mechanism of deformation welding. One is the film theory and the other is the energy barrier theory. As per the former, if two clean surfaces are brought into intimate contact, a weld will result. The latter proposes that an energy barrier exists which must be overcome before welding can result. The deformation processing of the joint serves to break up the film or overcome the energy barrier, depending on which theory one accepts. In any case, he observed that high quality, high reliability joints are produced when most alloys are carefully cleaned, brought into contact and subjected to deformation at elevated or room temperature. The major deformation welding variables were found to be degree of deformation, surface condition, composition, temperature, prior cold work and post-heat treatment. Gross plastic deformation, which promotes intimate contact and breaks surface oxides was found to be the principal factor in forming the weld in deformation welding. Degreasing followed by wire brushing was found to result in the best weld strengths with other parameters remaining the same. In general, alloying elements which increased the base metal hardness were found to increase the threshold deformation needed for deformation welding. Prior cold work was stated to have
little effect on the deformation welding of aluminum alloys.

2.5 Theories and mechanisms of diffusion welding

Cline (Ref. 22) considered the diffusion welding process to take place in two stages: (1) microscopic plastic deformation resulting in intimate metal-metal contact and (2) diffusion completing the weld and ultimately eliminating the interface. He listed the important diffusion welding variables as surface condition, time, temperature, pressure and the use of intermediate materials.

Ludemann (Ref. 14) listed necessary steps in the diffusion welding process as: (1) removing the oxide layer from the surface and preventing recontamination of the clean surface, (2) mating the surfaces under a sufficiently high mechanical force/temperature to bring the asperities into intimate contact and (3) the elimination of porosity along the weld line by diffusion. He discussed the theories that had been proposed for solid-state welding. He considered the evidence for the intimate contact or film theory and the energy barrier theory. He concluded that welding does occur upon intimate contact and the apparent activation energy seen
in diffusion welding is for removal of contaminants which prevent intimate contact rather than for the formation of metal-to-metal bonds. He excluded the mechanical or interlocking theory from consideration on the basis of experience with aluminum alloy bonding. Two soft aluminum pieces cleaned by a rotary brush, quickly placed together and given a blow were found to adhere together. But, if they were touched with fingers before being placed together, there was no welding, showing that the joint was not a result of mechanical interlocking.

Bartle (Ref. 16) described a diffusion weld as the result of microdeformation of surface asperities and diffusion across the interface. He listed a combination of similar and dissimilar metal joints among a variety of metals and alloys that were amenable for diffusion welding.

Garmong, Paton and Argon (Ref. 23) studied the diffusion welding of a Ti-6Al-4V alloy. The rate limiting step was found to be the complete elimination of porosity from the bond line. Welding was pictured as a two stage process: in the first stage, long wavelength surface asperities are flattened by plastic flow and in the second stage voids are reduced in size by a combination
of plastic flow and vacancy diffusion. Both stages were treated analytically and the results were used to predict welding times required. The microvoid collapse was modelled as the shrinkage of a thick walled hollow plastic sphere under hydrostatic pressure. The second mechanism considered for reducing the microvoid size was diffusion. The driving forces for the pore closure were pressure and surface energy. Long wavelength asperities were found to significantly increase welding time, while the short wavelength asperity height was not found to be as critical.

Derby and Wallach (Ref. 24) described a theoretical model for diffusion welding. They considered the various competing mechanisms that operate when two surfaces to be joined are brought together under applied pressure at an elevated temperature. The effect of the properties of the materials being joined and the process variables such as time, temperature and pressure on the competing mechanisms is discussed. The model is not specific to any metal or alloy and does not consider the effect of oxide films on the faying surfaces. The rate limiting step in the diffusion welding of copper was found to be the power law creep deformation of the original bond line surface roughness, allowing the two surfaces to come into
intimate contact.

The diffusion welding process was considered to be analogous to pressure sintering, although the geometry at the interface resulted in differences. The surfaces in contact were considered to consist of long triangular ridges and were assumed to contact peak to peak. Long voids or channels formed between the ridges which gradually shrank as the welding progressed. They postulated six welding mechanisms: (1) surface diffusion from surface sources to a neck, (2) volume diffusion from surface sources to a neck, (3) diffusion along the weld interface from interfacial sources to a neck, (4) volume diffusion from interfacial sources to a neck, (5) power law creep deforming the ridge and (6) plastic yielding deforming the ridge. The last mechanism was expected to occur only in the early stages of weding. Figure 5 shows a schematic view of the six mechanisms of mass transfer as the bond forms. Five process variables considered in the model were temperature, pressure, initial surface roughness, initial surface aspect ratio and time. A computer program incorporated material properties and rate equations for the six mechanisms mentioned above and could predict the dominant mechanism or the rate limiting step. For most combinations of process variables
Figure 5. Schematic view of a growing diffusion weld showing six different routes of mass transfer (Ref. 36)
resulting in the satisfactory diffusion welding of copper, the dominant mechanism was found to be power law creep.

Derby and Wallach (Ref. 25) improved on their earlier model by considering mass transfer in the vapor phase in addition to the aforementioned six mechanisms. This mechanism may be important in the diffusion welding of non-metals. In the earlier model, it was assumed that the channel between the ridges was of rhombic section. Since this cannot be the case if diffusional mechanisms simultaneously transport matter to a neck, the presence of a circular neck was provided for in the geometry calculations. They define Stage 0 of welding as an instantaneous plastic deformation of the contacting ridges until the local stresses are reduced to below the yield point of the material being welded. Stage 1 beginning with long prismatic interface voids continues until voids attain approximately circular cross-sections. Stage 2 then begins and throughout this stage the voids maintain their circular shape, while they shrink and finally disappear. On comparing their results with experimental results, they found that the changed model still required a change in geometry from Stage 1 to Stage 2 and that the transition in the geometry of the
voids needed to be reviewed.

An experimental study of the diffusion welding of de-oxidized copper was carried out by Derby and Wallach (Ref. 26). They also compared the results obtained with their theoretical model described above (Ref. 24). For contacting surfaces that had initial surface roughness values of 3.0 micrometers (120 microinches) mean height between peaks and valleys, the model predictions were found to be accurate at low welding temperatures and pressures. This was demonstrated by comparing a plot of fractional area welded versus temperature with a power law creep map, whereby the two plots coincided. The dominant welding mechanism was thus shown to be power law creep of contacting surface asperities. At higher temperatures and for very smooth initial surfaces, performance of the model was poorer.

Diffusion welds in iron and a low alloy steel (Ref. 27) showed the initial formation of the bond to be caused chiefly by surface diffusion along interfacial voids. Subsequent void closure occurred by other mechanisms. A comparison of machining with grinding as a surface preparation method for diffusion welding showed that although grinding produced a lower surface roughness that
was expected to aid welding, it was found to create a void pocket morphology that was detrimental to the removal of all residual porosity.

In summary, the models are limited by simplifying assumptions made on the geometry of interfacial ridges and voids. Agreements between predicted and observed rate limiting steps were found only in certain welding temperature ranges and for some starting surface finish values. The models appear to need more generalized treatment of interfacial void morphology and the transition in void morphology during successive stages of welding. However, long wavelength asperity height appears to more critically affect welding time than short wavelength asperity height.

2.6 Process Variables.

2.6.1 Characteristics of the metals being joined

Bryant (Ref. 28) proposed a method for specifying Hot Isostatic Pressure (HIP) welding parameters for many metals and alloys. Each material was classified into one of three categories based on its solubility for oxygen and the standard free energy of formation ($\Delta F^\circ$) of its most common oxide. Metals such as copper, tungsten and molybdenum, with high $\Delta F^\circ$ values greater than -100
KCal/mole, were placed in Category I of materials for which the least difficulty in preparing HIP welds due to the presence of an oxide layer would be expected. Materials such as titanium, tantalum and niobium, with intermediate $\Delta F^o$ values in the range -350 to -100 KCals/mole and solubilities for oxygen greater than 1.0 atom percent, were also included in this category. Materials with solubilities for oxygen less than 0.01 atom percent and $\Delta F^o$ less than -350 KCals/mole were placed in Category III. Materials in this category would be expected to exhibit the greatest resistance to HIP welding due to the presence of an oxide film. Materials with intermediate $\Delta F^o$ values (-350 to -100 KCals/mole) and intermediate solubilities for oxygen (0.01 to 1.0 atom percent) were placed in Category II. Aluminum, which falls in Category III, was found to require normalized welding temperatures (ratio of absolute welding to melting temperature) greater than 0.9 for any value of pressure. Thus, aluminum and its alloys are among the materials which exhibit the greatest resistance to solid-state welding due to the presence of an oxide film. As a result, if deformation during the bonding operation is to be precluded, it is necessary to replace the oxide layer with an element that dissolves and diffuses readily in aluminum and does not form undesirable microconstituents.
at the interface.

In comparing the deformation characteristics of different metals and alloy systems (Ref. 17), a general trend towards higher deformations being required with increasing melting point or hardness of the metal was observed. Hexagonal metals and their alloys were found to have considerably poorer welding properties than cubic metals as evidenced by higher threshold deformation at the same homologous temperatures. Alloying elements which increased the base metal hardness (Ref. 19) were found to increase the threshold deformation required for welding.

Golego (Ref. 29) studied the bonding of similar and dissimilar combinations of thirty different pure metals in high vacuum ($10^{-9}$-$10^{-10}$ mm of mercury). Surface films were cleaned in vacuum prior to the tests. Under a normal load of 0.05 times the tensile strength of the metal being tested, the change in the coefficient of friction as the metal pieces rubbed against each other was monitored. The strength of the bonds were taken as being reflected in the increase in the coefficient of friction. He found that metals with similar crystal lattices and slightly different atomic diameters (up to 13% different) tended to bond together. Metals such as cobalt and...
yttrium with very different atomic diameters (difference of more than 33%) did not tend to bond together. Dissimilar metal combinations such as iron-titanium, iron-cobalt and iron-zinc with different crystal structures and slightly different atomic diameters (up to 13% different) also tended to bond together. A pure metal in every case showed a tendency to bond to the same pure metal. The solubility of metals in each other did not affect the initiation or development of the bond process.

In a study of the roll bonding of dissimilar metals (Ref. 21), immiscible and slightly miscible systems were found to give stable welds. Some miscible metals were found to exhibit diffusional porosity upon heat treatment. With intermetallic forming systems in which the layer was brittle, the joint was strong if the layer was maintained below a critical thickness. The critical thickness depended on the small value of ductility in the intermetallic layer. If the interlayer was ductile and no porosity developed, the joint was strong and the failure occurred in the weaker metal.

A variety of properties of the base metal relevant to its solid-state joining were discussed in detail by Ludemann (Ref. 14). The properties considered were
ductility, elastic properties, melting point, crystal structure, orientation, texture, work hardening, phase changes, purity, oxygen solubility and surface energy. Ductility was found to have the greatest effect on bonding. Soft and ductile metals like indium were the easiest to join. This was attributed to a more ductile metal providing better contact for a given load. Regarding crystal structure, fcc metals bonded more strongly than bcc metals and both bonded more strongly than hcp metals. Metals which are capable of storing large amounts of elastic energy would be expected to show low values of measured bonding. This is due to the breaking of asperity junctions when the load was removed and elastic stresses were released. Lower melting point metals in general have greater ductility and hence bond together at lower values of deformation. The crystallographic orientation of the bond surface was not considered important for metallic materials, since in metals the bonds are not so directional. Diffusion welding was greatly enhanced for metals such as iron, copper and titanium which tend to dissolve their oxide films at high temperatures. Dissolution was stated to occur starting approximately at 0.5 $T_m$, where $T_m$ is the melting point in degrees Kelvin.
2.6.2 Welding Temperature

An increase in welding temperature generally aids the solid-state joining process through (1) improved deformation of surface asperities and better metal-metal contact owing to the lower yield strengths at higher temperatures, (2) enhanced diffusion, (3) desorption of surface impurities, whose thermal energy may exceed the bond energy as temperature increases, (4) an increased tendency to dissolve the oxide layer on the surface and (5) decreased elastic recovery resulting from the reduced elastic limit at higher temperatures. The threshold deformation was also found to decrease linearly with increasing temperature for the deformation welding of aluminum alloys 1100 and 1170 (Ref.30). Tylecote (Ref.24) reported the effect of increased bond temperature on the bond strength of deformation welded Al-Mg-Si alloy as shown in Figure 6.

Tylecote (Ref. 31) described diffusion welding as being carried out in the field of low pressure, high temperature and long times, where a reasonable degree of interdiffusion can take place rather than in the field of high pressure and short times, where considerable deformation occurs. But from a base material property standpoint, it is often desirable to limit the bonding
Figure 6. Bond strength as a function of deformation for aluminum alloys deformation bonded at elevated temperatures (Ref. 31)
temperature to as low a value as possible while still achieving a good bond. Base materials heat treated to a desired strength and toughness may tend to soften as the bonding temperature increases. It is also possible that very high bonding temperatures or lower temperatures coupled with high pressures may result in the need for structural support for the members being joined.

Barta (Ref. 32) presented a schematic representation of four different regimes or zones for diffusion welding aluminum alloys, as shown in Figure 7. Zone 1 requires no supporting tooling during the bonding operation or heat treatment of the bonded structure after welding. Zone 2 requires tooling, but no heat treatment. Zone 3 requires no tooling, but requires heat treatment and zone 4 requires both tooling and heat treatment.

Another consideration in choosing the welding temperature is the occurrence of phase transformations in the metal or alloy being welded, such as the alpha/beta transformation in a titanium alloy and the desirability or otherwise of the resulting microstructure. Overall, it appears that higher welding temperatures improve the ease with which one can achieve a satisfactory diffusion weld, but material property and tooling considerations make
Figure 7. Representative bonding parameter-zones for bonding a given design with an aluminum alloy (Ref. 32).
lower bonding temperatures desirable.

2.6.3 Welding Pressure

Ludemann (Ref. 14) describes the role of pressure in diffusion welding as needed to bring the faying surfaces together and high pressures as being applied mainly to aid in the closure of voids.

While modeling the diffusion welding of aTi-6 Al-4 V alloy, the primary experimental parameter affecting bond time was found to be the welding pressure at any given bonding temperature (Ref. 27). The weld time was defined as the time required such that no pore remaining in the interface had a size larger than 2000 Angstrom units (0.787 microroachines). With a long wavelength asperity height of 254 micrometers and a short wavelength asperity height of 2.5 micrometers, an increase of welding pressure from 3.45 MN/m (500 psi) to 34.5 MN/m (5 ksi) at a welding temperature of 927°C (1700°F) resulted in a reduction of the welding time from 12.0 hours to 0.7 hour. It was found to be important that no region along the bondline experienced locally low welding pressure.

Increasing welding pressure had a great effect on being able to reduce welding temperature without
sacrificing joint strength in the diffusion bonding of aluminum alloy 7075-T6. A threshold pressure was found to exist for each welding temperature (Ref. 32). This was the pressure above which the weld shear strength would not increase with increasing pressure and below which inferior or no appreciable bonding resulted. Up to 218 °C (425 °F), the threshold pressure was the compressive yield strength of the material. Above this temperature, a welding pressure of 20,000 psi, the compressive yield strength at this temperature was used.

Hamilton (Ref. 33) while considering the diffusion welding of a Ti-6Al-4V alloy, found that interfacial deformation under applied pressure was a necessary and important aspect of diffusion bonding. He predicted the pressure required to overcome interface asperities by creep deformation through an analysis utilizing average rates of strain and stresses resulting from applied pressure. The values predicted agreed favorably with reported data developed with a wide range of pressure, time and temperature combinations. The pressure requirements based on a plane-stress state assumption were approximately 70-85% of predictions based on a plane-strain state assumption. At bonding temperatures of 898-948 °C (1650-1740 °F), there was a sharp drop in
pressure as the time was increased to about 3 or 4 hours, but no change at further increased times.

Crane, Lovell, Baginski and Olsen (Ref. 34) studied the diffusion welding of aluminum alloy 2219 with type 321 stainless steel tubing. With a silver electroplated interlayer, they found that a bonding pressure that equalled or exceeded the compressive yield strength of the 2219 Aluminum alloy was needed at bonding temperatures of 260-315 °C (500-600 °F).

Cline (Ref. 22) reported the use of 7000 psi welding pressure while diffusion welding aluminum alloys at temperatures near 538 °C (1000 °F). With the use of a silver electroplate in the eutectic-diffusion welds between aluminum alloys, a pressure 300-500 psi was found to be sufficient.

Knowles and Hazlett (Ref. 47) investigated the diffusion welding mainly of beryllium and also of molybdenum, stainless steel, tungsten and commercially pure copper. The weld strength was found to increase linearly with welding pressure for silver plated beryllium at temperatures of 107, 138 and 204 °C (225, 280 and 400 °F) for pressures less than the compressive
yield strength of beryllium.

Dini, Kelley, Cowden and Lopez (Ref. 36) studied the use of electrodeposited silver as an aid in diffusion welding. They obtained a significant linear increase in joint strength with welding pressure at 300 °C (572 °F), but no significant effect at 600 °C (1112 °F) in the diffusion welding of stainless steel. They found that the pressure was relatively insignificant for beryllium and uranium. It is not clear from the data whether the bonding pressures were greater or less than the compressive yield strengths of the materials at the bonding temperature in each case.

O'Brien, Rice and Olsen (Ref. 37) studied the diffusion welding of silver-coated base metals. The tensile strength of silver coated Vascomax 250 maraging steel was found to increase significantly with bonding pressure in the range 10-20 ksi, remain nearly constant to about 30 ksi and then drop significantly with increasing pressure. A threshold welding pressure of 10 ksi was found necessary to achieve good contact at the silver-silver interface. The weld temperature was 204 °C (400 °F) and the weld time 10 minutes. The drop in bond strength beyond a welding pressure of 30 ksi was
attributed to the edges of the bonding surface starting to deform and promoting no-contact areas.

In summary, the bond strength appears to increase with bond pressure up to the compressive yield strength of the material at the bonding temperature. Above this pressure, the gains do not appear to be significant.

2.6.4 Welding time

In diffusion welding, particularly when the applied force is less than the yield strength of the base materials, creep and diffusion are required to achieve intimate contact. Thus, welding time would be expected to play an important role.

Once the surface asperities at the faying surfaces deform to generate a close contact, the welding time should be sufficient for the porosity at the weldline to disappear under the bonding conditions. For satisfactory strengths to be obtained, the metal must be able to dissolve surface films. It is also desirable to limit the time of growth of intermetallic layers at the interface, particularly if the layer is of a brittle nature.
Munir (Ref. 38) carried out a theoretical analysis of the stability of surface oxides during diffusion welding of metals. He classified metal oxides into three categories: unstable with respect to dissociation, unstable with respect to dissolution and stable. For a few metal oxides unstable with respect to dissociation, Table 2 shows the homologous temperature $T_h$ of instability with reference to the absolute melting point of the metal. If these metals are diffusion welded in air and if the temperatures are raised above the values shown above, welding between oxide free surfaces would be expected.

An oxide that is less stable as a phase than as a solution of oxygen in the metal was classified as unstable with respect to dissolution. The time necessary for complete dissolution of such an oxide depended on the ratio of the oxide layer thickness to the metal thickness. Metals Nb, Zr and Ti were found capable of dissolving thicker oxides than Ni, Co, Ta and Cu, regardless of the temperature.

For metals whose oxides were stable and whose oxide melting points were high, oxide-oxide sintering at normal
Table 2. Thermally unstable metal oxides (Ref. 38)

<table>
<thead>
<tr>
<th>oxide</th>
<th>$T_h$</th>
</tr>
</thead>
<tbody>
<tr>
<td>AgO</td>
<td>0.38</td>
</tr>
<tr>
<td>IrO</td>
<td>0.50</td>
</tr>
<tr>
<td>MoO</td>
<td>0.44</td>
</tr>
<tr>
<td>OsO</td>
<td>0.14</td>
</tr>
<tr>
<td>PdO</td>
<td>0.63</td>
</tr>
<tr>
<td>RuO</td>
<td>0.11</td>
</tr>
</tbody>
</table>
diffusion welding temperatures was considered highly unlikely.

In the diffusion welding of Alclad aluminum (Ref. 32), time was not found to be a critical variable in the range 1 to 4 hours. Actually some samples bonded for 1 hour exhibited higher shear strengths than samples bonded for 3 hours, other parameters remaining the same. The time of application of welding had no effect in small tool welding (Ref. 17).

The welding time in the range 15 to 240 minutes had a relatively minor influence on weld strength in the diffusion welding of aluminum alloys 1100 and 1170 (Ref. 19) bonded at a temperature of 482 °C (900 °F) without the use of any intermediate foils, when the deformation at the faying surfaces exceeded 15%.

Beryllium specimens bonded for times ranging from 0.5 to 240 minutes revealed that the bond strength was quite insensitive to bond time. This was substantiated by results from stainless steel (coated with 310 microinches of silver) and molybdenum specimens (Ref. 35).

In the diffusion welding of aluminum, uranium and
stainless steel, the effect of bonding time in the range 1 to 4 hours on bond strength was found to be insignificant (Ref. 36).

In summary, the time needed for the dissociation/dissolution of surface oxide layers/adsorbed impurities depends on the nature of the metal and its oxide. However, long welding times exceeding one hour were not found to significantly influence/improve bond quality in a variety of metals/alloys bonded.

2.6.5 Surface Finish

Surfaces to be diffusion welded are conventionally subjected to a bi-modal asperity analysis. Figure 8 shows waviness or long wavelength asperities spacing and roughness or short wavelength asperities spacing (Ref. 27). Typical surface roughness quantified by a CLA value of specimens prepared for diffusion welding at the Welding Institute is 0.4 micrometers (16 microinches). The extent to which rougher surfaces may be used was yet to be established. Surface waviness is not normally measured, but is expected to have a significant influence in the bonding of large surfaces (Ref. 39). In order to shorten the welding time, it was found desirable to keep the long wavelength asperity height to a minimum. It was
Figure 8. Characteristics of a surface being diffusion welded showing roughness and waviness

(Ref. 28)
found that the short wavelength asperity height was much less important than long wavelength asperity height so long as the latter was fairly small (Ref. 22).

In studying the use of electrodeposited silver as an aid in diffusion welding aluminum, stainless steel and uranium, no precautions were taken to obtain a specified surface finish on the faces to be joined. Typically, the finish was in the range 0.2 to 0.5 micrometers (8-21 microinches) of root mean square asperity height. (Ref. 36).

Typical Russian surface finish requirements were stated to be 0.2 micrometers (8 microinches). This type of surface finish would be expensive to achieve on production components and is one of the reasons why diffusion welding is used only when it shows technological advantages over other processes (Ref. 39).

2.6.6 The use of interlayers and their methods of deposition

The use of interlayers in the form of coatings or foils is often utilized in diffusion welding for a number of reasons such as (Refs. 22, 39, 40): (1) promoting plastic flow through the use of soft metals to generate
intimate contact, (2) providing a barrier to minimize undesirable intermetallics in dissimilar metal joints, (3) minimizing Kirkendall porosity, (4) obtaining clean surfaces, (5) reducing bonding temperature/time, (6) providing a smooth gradient of the coefficient of thermal expansion across a dissimilar joint, (7) causing temporary eutectic melting to scavenge the mating surfaces and promote diffusion of base metals and (8) replacing a very stable oxide layer on the surface of a metal like aluminum with an element that is soft and has a less stable oxide.

The use of silver as an interlayer in the diffusion welding of aluminum and its alloys is attractive owing to a variety of reasons. As seen from Table 2, the oxide of silver becomes thermodynamically unstable at a temperature of about 195 °C (384 °F). The use of almost any other interlayer material would require increased bonding temperatures and require vacuum/protective atmospheres during bonding.

Silver is a soft and ductile metal that promotes plastic flow at the interface to provide close matching of the faying surfaces. Such plastic flow would also be useful in promoting recrystallization at the interface in
the silver layer. The recrystallization temperature of cold-worked silver is about 200 °C (390 °F) and it was found that adherent silver-silver solid-state welds could not be made below this temperature even at pressures of 45,000 psi (Ref. 42).

Joints achieved with a thin intermediate layer are subject to restraint of plastic flow during tensile loading that result in a triaxial stress state at the joint. This prevents appreciable deformation in the joint and allows joint tensile strengths to be achieved that are many times the bulk ultimate strength of the intermediate layer material. O'Brien, Rice and Olsen (Ref. 37) showed that solid state welds in silver coated Vascomax 250 maraging steel coupons could be made with joint strengths as high as 110 ksi. This is against a bulk tensile strength of about 18.2 ksi in silver (Ref. 41).

Aluminum-stainless steel joints diffusion welded with 90 micrometers of silver on each substrate and heat treated at 280 °C (536 °F) for 192 hours were found to exhibit an intermetallic growth rate proportional to the square root of the time of heat treatment. The bulk of the intermetallic layer between silver and aluminum
consisted of \( \text{Ag}_2\text{Al} \), while a thin layer of \( \text{Ag}_3\text{Al} \) was observed in between the silver and \( \text{Ag}_2\text{Al} \) layers. The joint failed by a crack propagating through the \( \text{Ag}_2\text{Al} \) layer. During the diffusion welding process itself, only the \( \text{Ag}_2\text{Al} \) layer had formed.

Silver-aluminum composites rolled to 56% deformation at 540 \(^\circ\)C (1004 \(^\circ\)F) and held at this temperature for times up to 1 hour, did not show any reduction in bond strength with post-heat treatment (Ref. 26). The strength was nearly that of aluminum autogenously welded under similar conditions and the joint always failed in aluminum, although a substantial intermetallic layer developed at the interface. The stress-strain curves did not change in shape with thickness of the intermetallic layer, but the amount of plastic deformation that occurred before failure of the specimen was much greater where only thin intermetallic layers were present. An intermetallic layer thickness of 40 micrometers (0.0016 inch) was shown not to adversely affect the strength of the weld.

Hot Hollow Cathode Physical Vapor Deposition (HHCPVD) was shown to deposit extremely adherent coatings of silver on substrates such as aluminum and beryllium. The
coating process consisted of two steps: a high energy ion bombardment (cleaning phase) at chamber pressures below 10 torr, followed continuously by a low energy vapor deposition phase. The energy of bombarding ions during the cleaning phase is of the order of a few KeV making them capable of penetrating and removing contaminant layers (Ref. 44). This is in contrast to the process of physical vapor deposition, where evaporated atoms have energies of only a few-tenths of an electron volt and sputtering, where sputtered atoms have average energies of less than 10 eV (Ref. 45).

Cline (Ref. 22) found that 6061 aluminum alloy pieces diffusion welded after being silver coated by vapor deposition did not exhibit the weld integrity of identical test pieces that were electroplated. Knowles and Hazlett (Ref. 35), however, could obtain bond strengths of 40 ksi in beryllium, silver coated by the HHCPVD process and bonded at 107°C (225°F). The high strength bonds in vascomax steel referred to above (Ref. 31) had also been obtained with specimens silver coated using the HHCPVD process. The use of silver films evaporated by a HHCPVD process improved the diffusion bonding of aluminum to stainless steel (Ref. 43). High strength metallurgical bonds were reliably achieved
between the two at 204 °C (400 °F) and 16,969 psi pressure for 10 minutes. These parameters represented a significant improvement over the higher temperatures and longer times used in earlier studies. The improvements were attributed to the use of HHCPVD films rather than electroplated interlayers.

Dini (Ref. 40) could not find a simple explanation why HHCPVD coatings were superior to electroplated interlayers. Adhesion data on these coatings and electroplated layers on similar substrates showed comparable results (Ref. 46). He expected that with proper plating practice, electrodeposited coatings would be as good as HHCPVD coated films. However, he failed to take into account the effect of near surface defect density that may have been produced by the high energy cleaning step of the HHCPVD process. These defects may act as diffusion aids as well as promote more easy near surface deformation, promoting close contact at the interface.

2.6.7 Transient liquid phase bonding

The aluminum-silver eutectic occurs at a temperature of 566 °C (1050 °F) and a composition of 28 wt% aluminum as seen from Figure 9 (Ref. 6). Cline (Ref. 22) reported
Figure 9. Aluminum-Silver phase diagram (Ref. 6)
the accomplishment of diffusion welding of aluminum alloys with a closely controlled electroplate of silver, which produced a transient liquid phase above the silver-aluminum eutectic temperature. The liquid phase disperses the interfacial oxide layers and promotes atomic mobility to accomplish an aluminum diffusion weld with a silver rich zone adjacent to the weld line. Aluminum alloy 6061 electroplated with silver and eutectic-diffusion welded at 552 °C (1025 °F) for 15 minutes came apart during handling and increasing the holding time to 30 minutes did not improve the welding. The highest joint strength of 14-16 ksi was obtained with joints bonded at 580 °C (1075 °F) for 30 minutes. A silver thickness of nearly 0.0003 inch was found to be optimum for forming the eutectic layer and promoting bonding, but not leaving excess free silver at the interface.

Enjo and Ekeuchi (Ref.47) studied the diffusion welding of 2017 aluminum alloy in the temperature between the solidus and the liquidus of the alloy without the use of an interlayer. Differential Thermal Analysis (DTA) was used to determine the solidus of the alloy. Two to three percent by volume of liquid at a bonding temperature of 580 °C (1075 °F) gave the maximum bond strength of 270 MPa (39,160 psi), which increased to 400 MPa (58,015 psi) upon heat treatment. Larger volume fractions resulted in
porosity along the bond line. However, the tensile specimen was 7 mm (0.275 inch) in diameter at the gage length machined from the bonded specimen of 20 mm diameter.

In summary, it is seen that aluminum alloys are in general diffusion welded with a silver interlayer deposited by one of different methods. The weld quality and strength appear to improve with increasing welding temperature. Increased deformations at the faying surface result in virgin metal contact at the interface thereby improving bond quality, though a diffusion weld as strictly interpreted from its definition should be free from macroscopic deformation. Desirable welding pressures for diffusion welding aluminum alloys appear to be very close to the compressive yield strength of the alloy at the welding temperature.

Alloys with improved strength and creep resistance may be expected to need higher welding temperatures to complete the formation of a satisfactory weld since the closure of pores at the interface involves creep. Another factor inhibiting the formation of a solid-state weld in an Al-Fe type RS/PM alloy is the presence of finely dispersed and harder intermetallic dispersoids and grain-
to-grain hardness differences due to distribution of particulate cooling rates in the powder from which the alloy is fabricated. Faying surface cleaning procedures that remove the dispersoids and the use of higher welding temperatures than used for conventional alloys would be beneficial in this regard.
3.0 Objectives

The overall objective of this investigation was to study the diffusion welding characteristics of a rapidly solidified powder metallurgy Al-Fe-Ce alloy with silver interlayers and to compare the same with those of conventional ingot metallurgy aluminum alloys. Specific objectives included:

1. Performing short-term coarsening studies on the alloy to establish the effects of time, temperature and pressure of diffusion welding on the hardness and microstructure of the alloy and thereby develop diffusion welding parameter zones for the alloy that would permit its joining without excessive base metal degradation.

2. Developing effective silver deposition methods and produce welds using bonding parameter zones developed.
temperature gradients and specimen geometry as a means of increasing bonding pressures without increasing deformation at the faying surfaces.

4. Characterizing the diffusion welds to identify microstructural features that influence mechanical properties and fracture behavior and relate the observations to diffusion welding parameters.

5. Investigating higher temperature welding processes such as transient liquid-phase bonding, since the alloy is strengthened by dispersoids which do not dissolve in the alloy in the solid-state.
4. MATERIALS AND EXPERIMENTAL PROCEDURES

In this chapter, the fabrication process and the resulting microstructure of the Al-9Fe-4Ce alloy are discussed. Experimental procedures used in the study are described. They include a description of the equipment used and the reason for its choice, details of short-term coarsening studies to determine optimum bonding parameter zones for the alloy and specimen configurations used to study the effect of lateral constraints during bonding. Also included are cleaning and silver plating procedures used and details of diffusion welding studies with and without lateral restraint during bonding. Evaluation procedures employed for the joints are described including optical and scanning-electron microscopy, hardness testing, tensile testing, fractography and EDAX analysis.

4.1 Materials

An extruded 10 cm x 1.9 cm x 45.7 cm (4 inch x 3/4
designated as Al-9Fe-4Ce) alloy was obtained for this investigation. The extruded plate had been fabricated through flue-gas atomization of the melt and subsequent consolidation by vacuum hot pressing at 412 °C (775 °F) and extruding at the same temperature. This same alloy heat was used in all diffusion bonding studies. Short-term coarsening studies were conducted using another plate of Al-8.8Fe-3.7Ce alloy processed similarly. A few samples of the 514066-B4 were also subjected to coarsening studies to ensure a close similarity in coarsening characteristics between the two heats. The chemical compositions of the Al-Fe-Ce alloys are given in Table 3.

Figure 10a shows a scanning-electron micrograph of the flue-gas atomized Al-Fe-Ce powder. A wide range of powder particle sizes (5-40 micrometers) is seen to exist resulting in a wide range of particulate cooling rates during solidification (Ref.46). An optical micrograph of particle cross-sections is shown in Figure 10b. The small particles which cool relatively fast are seen to exhibit featureless microstructures designated in literature as Zone A (Ref.47). The larger particles which cool relatively slowly are seen to exhibit dark etching microstructures classified in literature as Zone B. In
Figure 10a. Scanning-electron micrograph of flue-gas atomized Al-Fe-Ce powder.
Figure 10b. Optical micrograph of cross-sections of Al-Fe-Ce powder showing light-etching Zone A and darker etching Zone B solidified microstructures, Keller's Etch.
the Al-9Fe-4Ce powder, both Zone A and Zone B microstructures solidified as primary alpha (face centered cubic solid-solution in aluminum) in a cellular mode, with the zone B contrast resulting from a coarser cell structure (Ref. 46).

Figures 11a and 11b show optical and scanning-electron micrographs of the as-received Al-9Fe-4Ce alloy. Decomposed Zone A regions are seen to etch light and some texture is observed in the direction parallel to the extrusion direction. The light-etching rounded particles in Figure 11a solidified with Zone A microstructures. These particles as mentioned were nearly twice as hard as those that solidified as Zone B (Ref. 9) due to supersaturation with solute elements. Consequently, they did not deform appreciably during consolidation of the powder and are seen in the figure as light-etching rounded particles. The light-etching streaks were relatively larger particles which solidified with Zone A or mixed Zone A and B microstructures. These were not quite as hard as the small Zone A particles mentioned above and elongated during extrusion. Their relatively high retained solute content with respect to particles that solidified as Zone B resulted in their light etching response. The microstructure of the alloy is complex and
Table 3. Chemical composition of Al-9Fe-4Ce alloy used in the diffusion bonding studies

<table>
<thead>
<tr>
<th>Element</th>
<th>Fe</th>
<th>Ce</th>
<th>Si</th>
<th>Mg</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>weight %</td>
<td>8.6</td>
<td>3.8</td>
<td>0.06</td>
<td>0.01</td>
<td>0.01</td>
</tr>
</tbody>
</table>
Figure 11a. Optical micrograph of as-received Al-9Fe-4Ce base metal, Kellers Etch. Arrows indicate particles that exhibited Zone A microstructures upon solidification and did not deform during thermomechanical processing of the powder.
Figure 11b. Scanning-electron micrograph of as-received Al-9Fe-4Ce base metal, Keller's Etch.
is known to contain a variety of fine intermetallic dispersoids as described earlier (Ref. 11)

4.2 Experimental Procedures

4.2.1 The GLEEBLE 1500 System

The GLEEBLE 1500 was chosen for diffusion bonding studies since it provided easy control of the time-temperature-pressure conditions used for bonding. It is an instrument manufactured by Duffers Scientific Inc., Troy, New York. It can subject a sample to programmed thermal and mechanical cycles, each of which can be controlled independently. Jaws and inserts usually made of a copper alloy or austenitic stainless steel are used to grip the specimen. Thermocouples percussion welded to the specimen at the center of its length monitor its temperature. The specimen is resistance-heated by a large alternating current. Since alternating current is used to heat the specimen, electro-transport effects, if any, during the positive and negative halves of each cycle annul each other. Due to temperature gradients along the length of the specimen only the section of the specimen near the thermocouples experiences the programmed thermal cycle. A servo-hydraulic system controls the mechanical cycle. The control parameter may be the force, stroke or the strain that a specimen is subjected to. The specimen
chamber can be evacuated or backfilled with an inert gas as desired. The GLEEBLE was used for all the diffusion welding studies carried out in this investigation. A view of the GLEEBLE and specimens assembled for bonding is shown in Figures 12, 13 and 14. Elastic constraint effects due to the temperature gradients along the length of the specimens being bonded permit the use of increased bonding pressures without increased macroscopic deformation. The bonding chamber atmosphere can be relatively easily controlled. However, the control of small values of pressures used in the transient-liquid phase bonding is more difficult.

4.2.2 Selection of GLEEBLE jaws and inserts used in bonding

4.2.2.1 Use of copper jaws and copper inserts

The use of water cooled copper jaws with copper inserts created steep temperature gradients of a few hundred degrees centigrade per centimeter along the length of the samples being bonded by effectively cooling one end of the sample to room temperature. The faying surfaces were at the center of the distance between the inserts.
Figure 12. The GLEEBLE 1500 and the personal computer through which specimen thermal and mechanical cycles may be programmed.
Figure 13. A view of the specimen chamber in the GLEEBLE.
Figure 14. An assembly of specimens to be bonded using copper jaws and stainless inserts (inserts are not seen in the picture).
and were maintained at the programmed temperature by the GLEEBLE. The other ends of the samples which were in contact with the copper inserts were essentially at room temperature. The resultant elastic constraint effects operating at the faying surfaces tended to restrain them from lateral deformation even when the applied pressure exceeded the yield strength of the material being bonded at the bonding temperature. The magnitude of the constraint effect depended on the temperature gradient along the sample, which in turn depended on the span length between the inserts. Samples of 2024-T6 aluminum alloys could be subjected to 2-3 times the yield strength at temperature depending on specimen span length between the jaws without any macroscopic deformation occurring at temperatures up to 300 °C (572 °F) for times up to 2 minutes. The loads applied on the samples were found to be accurate to within 2-3% of the values recorded using calibrated load cells. The beneficial effect of increased pressure in diffusion bonding has been clearly brought out in Section 2.4.3 with respect to the diffusion welding of a number of metals and alloys. Creating steep temperature gradients in a direction transverse to the faying surfaces can thus significantly reduce bonding times/temperatures by enabling the application of much higher pressures than the yield
strength of the alloy without any macroscopic deformation occurring. At any welding temperature, the increased applied pressure should help in pore closure at the interface and reduce welding time. Alternately, it may be possible to reduce the welding temperature for a given welding time.

4.2.2.2 Use of copper jaws with stainless steel inserts

The temperature gradients along the length of a sample were much shallower with this combination of sample holding fixtures, on the order of a few 10s of degrees centigrade per centimeter. Thus, the benefits of constraint effects permitting the use of increased bonding pressures were minimal. This combination was chosen for the production of welds in this study in order to minimize risks of overheating the GLEEBLE. In this case, the copper jaws were again water cooled and were in close contact with the specimen holding type 304-L stainless steel inserts.

4.2.2.3 Stainless steel "hot" jaws

In this case, split-stainless steel jaws with threads matching those on the specimen were used to hold the specimens being bonded. The jaws have many holes drilled in them to minimize the heat drawn into the machine from
the sample. Thus lower resistance heating currents are required to maintain the sample temperatures during the bonding period. Very shallow temperature gradients were created along the sample and there was no risk of overheating the GLEEBLE during even long bonding periods. Also, the design of the jaws made necessary the use of specimens which were at least 3 cm (1.2 inches) long. The specimens tended to sag or buckle during a bonding operation owing to the shallow temperature gradients and long specimen lengths. Consequently, they were not used in this study.

4.2.3 Coarsening Studies

The purpose of the coarsening studies was to evaluate the effects of temperature, pressure and time of diffusion welding on the microstructure and hardness of the alloy. Earlier studies on coarsening of the alloy (Ref. 42) had been restricted to long periods of time ranging from 24 hours to 2,160 hours which may be more relevant to the coarsening characteristics of the alloy during service at high temperatures than during diffusion bonding. Data on the effect of temperature during shorter diffusion welding times was not available and needed to be generated. From this data it would be possible to predict the decrease in hardness of the alloy during any
time-temperature-pressure regime of diffusion welding that may be developed. Also, if the decrease in hardness during diffusion welding is to be limited to any desired value, the time-temperature-pressure regimes within which it would be possible to do so could be established.

4.2.3.1 Heat Treatment in a Tubular Furnace

Square bars of cross-section 2.54 cm X 2.54 cm (1 inch X 1 inch) were cut from the as-received Al-9Fe-4Ce extrusion with the length of the bar along the extrusion direction. Slices 2.54 cm X 2.54 cm X 0.16 cm (1 inch X 1 inch X 1/16 inch) were cut from these bars and were heat treated in a tubular furnace at temperatures ranging from 300 to 500 °C (572 to 932 °F) for times ranging from 5 minutes to 1 hour. The furnace was heated to the desired temperature and the sample was introduced. No pressure was applied during heat treatment. A chromel-alumel thermocouple welded to the center of a sample was used to ensure that the temperature read on the furnace meter was accurate to within 2-3 °C (3.6 to 5.4 °F). On monitoring the thermocouple output, it was observed that in this temperature range, the sample reached the furnace temperature in approximately 4 minutes. The time of heat treatment of each sample recorded was the time of soaking after the initial 4 minutes. The heat treated samples
were sectioned, mounted, polished, etched in Keller's reagent and observed in optical and scanning-electron microscopes. The sections were also subjected to Vicker's microhardness testing.

4.2.3.2 Heat Treatment in the GLEEBLE 1500

Rectangular bars 1.27 cm \times 1.9~cm (0.5 inch \times 0.75 inch) in cross-section were cut from the as-received Al-9Fe-4Ce extrusion with the length of the bar parallel to the extrusion direction. Pieces 1.27 cm (0.5 inch) in length were cut from these bars. Each piece was heat treated in the GLEEBLE 1500 using stainless steel inserts. The stainless steel inserts were 1.0 inch in diameter and were held coaxially in the stationary and moving jaws of the GLEEBLE. Chromel-alumel thermocouples welded to the center of each specimen were used to monitor the temperature of the specimen through the heat treatment cycle. The extrusion direction of the plate was transverse to the direction of application of force during heat treatment. Each sample was heated to temperature in 2 seconds, held at temperature for periods of time ranging from 1 to 12 minutes and water quenched. The heat treatment temperatures used were 400-560 °C (752-1040 °F). Compressive pressures applied were up to and beyond the yield strength of the alloy at the heat
treatment temperature. Cross-sections of heat treated samples at thermocouple locations were cut using a Buehler Isomet low speed cutting saw. These sections were subjected to optical and scanning-electron microscopy and microhardness testing as per procedures in Section 4.2.3.1.

4.2.3.3 Differential-Scanning Calorimetry

Samples weighing 20 to 25 milligrams were sliced from the Al-9Fe-4Ce plates using a Buehler low speed saw. These were cleaned in methyl alcohol and subjected to Differential Scanning Calorimetry (DSC) using a Perkin-Elmer DTA 1700 unit with a Data Station 3600. The accuracy of temperature measurement by the unit was checked by measuring the melting points of 99.99 percent pure aluminum and 99.99 percent pure nickel. The enthalpy measurements in the unit were calibrated using the measured enthalpy of fusion of pure aluminum. Base line calibration of the unit was carried out using a pure alumina standard sample. A scan rate of 20°C (36°F) per minute was used in all runs. Argon flow at the rate of 20 milliliters per minute was maintained over the specimen holding cups during each run. Each sample was scanned on heating from room temperature to 720°C (1328°F). A change of slope in the DSC plots obtained on heating was used to
determine the onset of microstructural coarsening. The solidus of the alloy was determined from the onset of melting in the alloy during the heating run.

4.3 Diffusion Bonding

4.3.1 Specimen Geometry

Cylindrical specimens 1 cm (0.394 inch) long and with 1 cm diameter metric threads at one end were machined from the Al-9Fe-4Ce extrusion with the length of the specimen oriented parallel to the extrusion direction. The threads matched those on the GLEEBLE hot jaws and threads in the electro-plating fixtures. The other end of the specimen was machined to generate specimen sets for diffusion welding as shown in Figure 15. Type A specimens were straight cylinders with the bonding faces machined square in a lathe. Type B specimens were machined to produce a cylinder and cup fit to provide lateral constraint during diffusion welding. These were machined with two different values of diametral clearance between the cylinder and the cup and were designated B1 and B2. Type B1 specimens were machined with a tight fit clearance between the mating parts not exceeding 0.008 cm (0.003 inch) in diameter. Type B2 specimens were machined with a diametral clearance between mating parts so that the area of the cup was 5% in excess of the inner
Type A-Straight Cylinders

Type B-Cylinder and cup fit for lateral restraint

Figure 15. Specimen configurations used in diffusion welding.
specimen's area. Type B1 specimens were designed to avoid any deformation at the faying surfaces during diffusion welding and Type B2 specimens were designed to limit deformation to 5% as measured by the increase in area at the faying surfaces.

4.3.2 Specimen Cleaning Procedures

Standard cleaning procedures recommended for aluminum alloys (Ref. 43) as shown in Table 4 were used. The purpose of the cleaning procedures was to remove any dirt, grease, foreign matter or oxide scales from the faying surfaces and to produce a surface with a thin oxide layer of uniform activity for zincating and electroplating operations. The dispersoids in the alloy are hard and consist of elements which have a very low diffusion coefficient in the aluminum matrix. Hence, it would be desirable to remove these as well from the faying surfaces during the cleaning procedures and to expose the softer face-centered cubic alpha phase at the faying surfaces to be electroplated for bonding. Etching in 25% sulfuric acid removed the dispersoids from the surfaces being cleaned. The effectiveness of the cleaning procedures was judged by visually examining the surface after each cleaning step for uniformity of appearance and the time taken for zinc to deposit and the
Table 4. Cleaning procedures used for Al-9Fe-4Ce alloy

Degrease specimen in acetone with ultrasonic agitation

↓

Clean in 22 grams/liter Na$_2$CO$_3$ + 22 grams/liter Na$_3$PO$_4$
(Solution A) at 80°C (176°F) for 2-3 minutes and rinse in distilled water

↓

Clean in 50% water + 50% HNO$_3$ by volume with a few drops of HF (Solution B) and rinse in distilled water

↓

Clean in 25% by weight H$_2$SO$_4$ in water (Solution C) at 80°C (176°F) for 2-3 minutes and rinse in distilled water

↓

Clean in 50% water + 50% HNO$_3$ by volume with a few drops of HF and rinse in distilled water

↓

Zincate for 30 seconds by immersing in a zincating solution containing 100 g/l ZnO, 525 g/l NaOH, 9.8 g/l Rochelle Salt and 1.0 g/l FeCl$_3$ dissolved in water (Solution D)

↓

Rinse in water with ultrasonic agitation and dry
uniformity of its deposition during the subsequent zincating step. Between the cleaning and zincating steps, the first few specimens were observed under a stereo-microscope at 10-70 X magnification to check whether the dispersoids had been removed from the cleaned surfaces.

4.3.3 Silver plating procedures

The cleaned samples were coated with micromask compound from Pyramid Plastics Inc., Arkansas on all surfaces except the faying surfaces and the threads that would be mounted on to matching nuts in the electroplating fixture. All parts of the electroplating cathode fixture that were immersed in the plating solution were also coated with this compound. This was for selective masking of all areas on which plating was not required in order to maintain a close control over the current density during plating. The micromask compound was allowed to dry overnight. Immediately prior to electroplating, the zinc coating on the surfaces to be plated was dissolved in Solution B mentioned in Table 5 and rezincated in Solution D for 30 seconds with ultrasonic vibration during zincating. The zinc layer was again dissolved in solution B and redeposited with Solution D for 30 seconds with ultrasonic agitation. The zincating solution D dissolves the oxide layer on the
aluminum surface. As soon as metallic aluminum is exposed to the zincating solution, a displacement reaction occurs, dissolving aluminum and depositing zinc. Ultrasonic vibration during the zincating step helps in producing an adherent zinc coating by dispersing any loosely adhering deposit and recoating with an adherent deposit (Ref. 44). The zincated samples were ultrasonically cleaned in distilled water and immediately mounted on to the electroplating fixture. A 99.9% pure silver anode was used with the solutions mentioned in Table 6 for silver plating. Zincated surfaces were not allowed to become dry prior to silver strike plating. An initial silver strike for 30-60 seconds was used to obtain a uniform silver layer on the faying surfaces, followed by electroplating for 30 minutes. Current densities used during plating are as given in Table 5. The plated specimens were ultrasonically cleaned in acetone to remove the micromask compound, in distilled water, and finally in methyl alcohol, dried and stored in a desiccator. Samples electroplated for different times were sectioned, mounted, polished and observed in an optical microscope to determine the plating thickness and to qualitatively evaluate the adhesion of the plating.
Table 5. Silver plating solutions and conditions

<table>
<thead>
<tr>
<th>A. Silver-Strike Solution</th>
<th>B. Silver Plating Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.3 grams/liter AgCN +</td>
<td>45 grams/liter AgCN +</td>
</tr>
<tr>
<td>67.5 grams/liter NaCN</td>
<td>65 grams/liter KCN +</td>
</tr>
<tr>
<td>Dissolved in distilled water</td>
<td>50 grams/liter $\text{K}_2\text{CO}_3$</td>
</tr>
<tr>
<td>Current density 2A/dm$^2$</td>
<td>Current density 1 A/dm$^2$</td>
</tr>
</tbody>
</table>
4.3.4 Establishment of welding regimes and optimum welding parameters

An important aspect of this work was the generation of diffusion welding time-temperature-pressure regimes for the dispersion strengthened aluminum alloys. Samples for diffusion welding of types A, B1 and B2 were machined, cleaned as per procedures in Section 4.3.2, zincated for 30 seconds, silver strike plated and silver plated for 30 minutes for 30 minutes as per procedures in Section 4.3.3. The plated samples were ultrasonically cleaned in methyl alcohol prior to the welding operation. Chromel-alumel thermocouples were percussion welded to the samples on their cylindrical surfaces within 1 mm (0.039 inch) of the faying surfaces to monitor their temperature during the bonding cycle. The specimens were mounted in the GLEE BLE using copper jaws and stainless steel inserts. The specimen chamber was closed and evacuated to a pressure of 100 milli-torr. It was backfilled with argon gas and evacuated to 100 milli-torr pressure.

The specimens were bonded at temperatures ranging from 350 to 500 °C (572 to 932 °F). Bonding time-temperature-pressure combinations used were based on bonding parameter zones that were developed and which are described later.
4.3.5 Transient liquid-phase bonding

Specimens to be transient-liquid phase bonded were prepared in a similar way except that they were not zincated prior to silver-strike plating. Electroplating time was limited to 10 minutes if a plated sample was bonded with a non-plated sample and 5 minutes if both samples being bonded were plated. The specimens were bonded at temperatures ranging from 550 to 635 °C (1022 to 1175 °F). Bonding time-temperature-pressure combinations used were again based on bonding parameter zones that were developed and are described later. The bonded samples were evaluated in a way similar to the diffusion-bonded samples and described later in sections 4.3.6.1 to 4.3.6.4.

4.3.6 Evaluation of the bonds

4.3.6.1 Optical and scanning-electron microscopy

Longitudinal diametral sections of as-bonded specimens were cut using a Buehler saw, mounted and prepared for microscopic examination. The mounted sample was ground, polished in alumina, fine-polished using Buehler Mastermet polishing suspension and etched in Keller's reagent. The prepared specimens were observed in a PFME Olympus metallograph and in a Hitachi S-510 scanning-
electron microscope. EDAX analysis was performed in a JEOL JXA-35 scanning-electron microscope.

4.3.6.2 Microhardness testing

Microhardness traverses were taken across the bonds in the above mounted longitudinal sections of bonded specimens in a Buehler Micromet microhardness tester with a diamond pyramid indentor at a load of 500 grams. A single reading was reported for each point of the hardness traverse made across the diffusion-welded region.

4.3.6.3 Tensile testing

Sheet specimens for tensile testing that consisted of longitudinal slices about 2 mm (0.078 inch) thick were cut from welded specimens using a Buehler low speed saw. The specimen widths were reduced by 30-40% from original specimen widths to avoid edge effects during diffusion welding from influencing test results. These were tensile tested in an Instron 4204 tensile test machine to determine the bond strength. The pull-out load in each case was noted to determine the bond strength.

4.3.6.4 Fractographic examination

Fractured surfaces of tensile test coupons were
subjected to fractographic examination in a Hitachi S-510 scanning electron microscope. EDAX analysis was performed with a JEOL JXA-35 scanning-electron microscope equipped with a Kemp x-ray spectroscopy system.
5. Results and Discussion

5.1 Coarsening Studies

Vicker's pyramidal hardness numbers (VPN) using a 500 gram load on as-received and tubular furnace heat treated samples are shown in Table 7. Each of the hardness values is an average obtained from two indentations. Some of the hardness values are plotted as functions of heat treatment time and temperature in Figure 16. It is seen from Table 7 and Figure 16 that samples heat treated at temperatures above 412 °C (775 °F), the hot pressing and extrusion temperature of the alloy, showed effects of some secondary hardening probably due to the formation of complex precipitates/dispersoids.

The hardness values of samples heat-treated in the GLEEBLE are plotted as a function of time and temperature in Figure 17. The compressive stress applied to each sample during heat treatment was just sufficient to hold the sample between the jaws and did not exceed 1000 psi. Samples discussed in Figure 17 did not yield during heat treatment. However, a small difference in time scales
between Figures 16 and 17 should be recognized as the GLEEBLE heat-treated samples are heated to temperature in a few seconds, while the furnace samples took several minutes to reach the heat-treatment temperature. The estimated time of 4 to 5 minutes to reach temperature is not included in the time shown against the furnace heat treated samples. Consequently, 10 minutes of furnace heat-treatment should correspond to about 15 minutes of heat-treatment in the GLEEBLE. Since the time-axis in Figure 17 does not extend to 15 minutes, it does not show the secondary hardening effects observed in Figure 16.

Table 8 presents hardness data on GLEEBLE heat treated samples for which the applied compressive stress during heat treatment was varied. It is seen that at a temperature of 500 °C (932 °F) the applied pressure did not have a significant effect on the hardness values except when the sample deformed plastically. At 450 and 400 °C (842 and 752 °F) the effect of pressure was not so clear as the hardness values were scattered in a range of 10 VPN. In all cases, a change in applied pressure over a wide range was found to result in a hardness change of 10 VPN or less.
Table 7. Vicker's hardness values of as-received and tubular furnace heat treated samples

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Treatment Temperature (°C)</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>40</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>As recd.</td>
<td>131.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>300</td>
<td>131.7</td>
<td>131.7</td>
<td>129.5</td>
<td>128.4</td>
<td>116.3</td>
</tr>
<tr>
<td>3</td>
<td>350</td>
<td>130.7</td>
<td>129.7</td>
<td>127.9</td>
<td>128.3</td>
<td>127.3</td>
</tr>
<tr>
<td>4</td>
<td>375</td>
<td>125.5</td>
<td>124.8</td>
<td>126.2</td>
<td>121.8</td>
<td>125.3</td>
</tr>
<tr>
<td>5</td>
<td>400</td>
<td>130.9</td>
<td>130.2</td>
<td>127.3</td>
<td>121.6</td>
<td>128.5</td>
</tr>
<tr>
<td>6</td>
<td>450</td>
<td>128.0</td>
<td>129.8</td>
<td>123.0</td>
<td>116.2</td>
<td>120.9</td>
</tr>
<tr>
<td>7</td>
<td>475</td>
<td>126.6</td>
<td>120.8</td>
<td>124.6</td>
<td>128.5</td>
<td>121.4</td>
</tr>
<tr>
<td>8</td>
<td>500</td>
<td>116.2</td>
<td>123.9</td>
<td>112.3</td>
<td>97.1</td>
<td>99.6</td>
</tr>
</tbody>
</table>
Figure 16. Vicker's hardness of tubular furnace heat-treated Al-9Fe-4Ce samples.
Figure 17. Vicker's hardness of GLEEBLE heat-treated Al-9Fe-4Ce samples as a function of time and temperature of heat treatment.
Table 8. Vicker's hardness of GLEEBLE heat treated samples with applied pressure as a variable.

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Temperature (°C)</th>
<th>Time (minutes)</th>
<th>Pressure (psi)</th>
<th>VPN</th>
<th>Whether sample yielded</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>500</td>
<td>10.0</td>
<td>2500</td>
<td>123.6</td>
<td>No</td>
</tr>
<tr>
<td>23</td>
<td>500</td>
<td>10.0</td>
<td>5000</td>
<td>113.1</td>
<td>Yes</td>
</tr>
<tr>
<td>24</td>
<td>500</td>
<td>5.0</td>
<td>1120</td>
<td>127.2</td>
<td>No</td>
</tr>
<tr>
<td>25</td>
<td>500</td>
<td>5.0</td>
<td>2020</td>
<td>125.6</td>
<td>No</td>
</tr>
<tr>
<td>26</td>
<td>500</td>
<td>5.0</td>
<td>3030</td>
<td>127.3</td>
<td>No</td>
</tr>
<tr>
<td>27</td>
<td>500</td>
<td>5.0</td>
<td>3815</td>
<td>125.5</td>
<td>No</td>
</tr>
<tr>
<td>28</td>
<td>500</td>
<td>5.0</td>
<td>5050</td>
<td>122.7</td>
<td>Slightly</td>
</tr>
<tr>
<td>29</td>
<td>500</td>
<td>5.0</td>
<td>6510</td>
<td>120.5</td>
<td>Yes</td>
</tr>
<tr>
<td>30</td>
<td>450</td>
<td>5.0</td>
<td>900</td>
<td>135.0</td>
<td>No</td>
</tr>
<tr>
<td>31</td>
<td>450</td>
<td>5.0</td>
<td>3140</td>
<td>126.5</td>
<td>No</td>
</tr>
<tr>
<td>32</td>
<td>450</td>
<td>5.0</td>
<td>5400</td>
<td>128.3</td>
<td>No</td>
</tr>
<tr>
<td>33</td>
<td>450</td>
<td>5.0</td>
<td>7850</td>
<td>133.6</td>
<td>Slightly</td>
</tr>
<tr>
<td>34</td>
<td>450</td>
<td>5.0</td>
<td>10550</td>
<td>130.7</td>
<td>Yes</td>
</tr>
<tr>
<td>35</td>
<td>400</td>
<td>5.0</td>
<td>900</td>
<td>134.7</td>
<td>No</td>
</tr>
<tr>
<td>36</td>
<td>400</td>
<td>5.0</td>
<td>4150</td>
<td>131.7</td>
<td>No</td>
</tr>
<tr>
<td>37</td>
<td>400</td>
<td>5.0</td>
<td>5500</td>
<td>130.1</td>
<td>No</td>
</tr>
<tr>
<td>38</td>
<td>400</td>
<td>5.0</td>
<td>7750</td>
<td>125.6</td>
<td>No</td>
</tr>
<tr>
<td>39</td>
<td>400</td>
<td>5.0</td>
<td>11000</td>
<td>131.7</td>
<td>Slightly</td>
</tr>
</tbody>
</table>
Figure 18 permits selection of time-temperature regimes of diffusion welding that result in desired maximum amounts of softening (reduction in hardness) of the base metal. All time-temperature combinations below curve A result in a base metal softening of less than 10 VPN. Those between curves A and B result in softening by 10-20 VPN and those above curve B by more than 20 VPN. The effect of interlayer material diffusion into the base alloy during welding on base metal hardness is of course not considered in the above treatment.

5.2 Differential Scanning Calorimetry

Figure 19 shows a DSC plot of the Al-9Fe-4Ce alloy at a scan rate of 20 °C (36 °F) per minute. The change of slope seen at a temperature slightly more than 400 °C (752 °F) is shown on an expanded scale in Figure 20. The change of slope is seen to occur at nearly 420 °C (788 °F). At this scan rate, the coarsening of the alloy microstructural features/formation of new precipitates or dispersoids appears to begin at this temperature. This agrees favorably with the fabrication history of the alloy, since it was hot pressed/extruded at 412.8 °C (775 °F). At the scan rate employed in the DSC runs, changes in microstructural features are not expected below the extrusion temperature of the alloy, as such changes would
Figure 18. Diffusion-welding parameter zones for the Al-9Fe-4Ce alloy. Time-Temperature combinations on or below curve A result in softening of 10 VPN or less and Curve B 20 VPN or less.
Figure 19. DSC plot of Al-9Fe-4Ce alloy.
Figure 20. DSC plot of Al-9Fe-4Ce alloy in the temperature range 300 to 600°C on an expanded scale.
Figure 21. DSC plot of Al-9Fe-4Ce alloy in the temperature range 600 to 720 °C on an expanded scale.
have taken place during the extrusion process itself. Figure 21 shows the portion of Figure 19 beyond 600 °C (1112 °F) in an expanded scale. The solidus of the alloy corresponding to the initiation of melting is seen to be at 638 °C (1180.4 °F).

5.3 Microstructures of heat-treated Al-9Fe-4Ce samples

Figures 22 to 26 show scanning-electron micrographs of Al-9Fe-4Ce material heat treated at 300, 350, 400, 450 and 500 °C (572, 662, 752, 842 and 932 °F) for 5, 20 and 60 minutes in a tubular furnace. No observable coarsening of microstructural features was seen in samples heat treated at temperatures of 300 and 350 °C for times up to 60 minutes (Figures 22 and 23). Figures 24c (400 °C/60 minutes) and 25c (450 °C/60 minutes) show increased etching response in Zone A regions. This was due to the formation of increased amounts of equilibrium Fe and Ce containing dispersoids and Al₃Fe, as found in earlier studies (Ref.11). Coarsening is much more rapid at 500 C. Figures 26b and c show significant coarsening in Zone A regions and a general coarsening of the microstructure. Earlier studies indicated only equilibrium phases retained in samples heat treated in this range for two hours. Zone A regions in Figures 26b and c are seen to have "decomposed" to a large extent. The formation of
equilibrium $\text{Al}_3\text{Fe}$ and $\text{Al}_{10}\text{Fe}_2\text{Ce}$ at the expense of metastable phases should be near completion in the decomposed Zone A regions of these samples. The microstructures are more complex in the Zone B regions. The metastable phases $\text{Al}_5\text{Fe}$ and the Al-Fe-Ce phase with equal atomic proportions of Fe and Ce have probably transformed to the equilibrium phases $\text{Al}_3\text{Fe}$ and $\text{Al}_{10}\text{Fe}_2\text{Ce}$, and the transformation of the Al-Fe-Ce phase with an Fe/Ce atom % ratio of four to equilibrium phases may have proceeded partially.

The above interpretation of microstructural changes based upon previous work on the alloy (Ref.11) is considered adequate for this work, as the intention is to keep microstructural changes to a minimum during the diffusion welding regimes employed and to identify time-temperature combinations of diffusion welding that permit the same. Increased etching response of the features in the decomposed Zone A regions is not seen in samples whose hardness decrease upon heat-treatment did not exceed 10 VPN (Figures 22 a, b and c, 23 a, b and c, 24 a and b and 25 a). Thus, the onset of observable increased etching response of the decomposed Zone A regions appears to occur for heat-treatment time-temperature combinations on or slightly above line A in Figure 18.
Figure 22. Scanning electron micrograph of Al-9Fe-4Ce alloy heat treated at 300°C (572°F) for A) 5 minutes; B) 20 minutes; C) 60 minutes. Keller's Etch.
Figure 23. Scanning electron micrograph of Al-9Fe-4Ce alloy heat treated at 350°C (662°F) for
A) 5 minutes; B) 20 minutes; C) 60 minutes.

Keller's Etch.
Figure 24. Scanning electron micrograph of Al-9Fe-4Ce alloy heat treated at 400\(^\circ\)C (752\(^\circ\)F) for A) 5 minutes; B) 20 minutes; C) 60 minutes. Keller's Etch.
Figure 25. Scanning electron micrograph of Al-9Fe-4Ce alloy heat treated at 450°C (842°F) for
A) 5 minutes; B) 20 minutes; C) 60 minutes.
Keller's Etch.
Figure 26. Scanning electron micrograph of Al-9Fe-4Ce alloy heat treated at 500°C (932°F) for A) 5 minutes; B) 20 minutes; C) 60 minutes. Keller's Etch.
5.5 Diffusion Welding Parameter Zones for the RS/PM Al-Fe-Ce alloy

Figures 27 a and b present welding parameter zones for the RS/PM Al-9Fe-4Ce alloy similar to those in Figure 7 for conventional aluminum alloys. The RS/PM alloy is not heat-treatable. For heat-treatable alloys the welding regimes are separated into those that do and do not require a post-weld heat treatment. Such separation is not relevant to the RS/PM alloy. As such, only two zones are applicable to the diffusion welding of the RS/PM class of alloys versus four zones for the conventional heat-treatable aluminum alloys. The boundary between the two zones in each figure separates welding regimes that result in 5-10% or less deformation at the faying surfaces from those that result in more than 10% deformation, with the time of welding at each temperature chosen based on coarsening studies from curve A of Figure 18. Figure 18 is reproduced below to make apparent the time of soaking at each temperature. Hence, if diffusion welding of the alloy is carried out using bonding regimes in Zone 2, the alloy will need tooling for structural support in directions transverse to the faying surfaces, if deformation is to be kept below 10%. The zones were established experimentally in the GLEE BLE using copper
Figure 27a. Representative bonding parameter zones for the RS/PM Al-9Fe-4Ce alloy using type A straight cylinder specimens. Zone 1 represents welding regimes that require no supporting tooling and Zone 2 regimes that require tooling.
Figure 27b. Representative bonding parameter zones for the RS/PM Al-9Fe-4Ce alloy using type B cylinder-cup specimens with lateral restraint. Zone 1 represents welding regimes that require no supporting tooling and Zone 2 regimes that require tooling.
Figure 18. Diffusion welding temperature-time parameter zones for Al-9Fe-4Ce alloy. Time-Temperature combinations on or below curve A result in base metal softening of 10 VPN or less and curve B 20 VPN or less.
jaws with stainless steel inserts to hold the specimens. Figure 27a was established using type A straight cylinder specimens and Figure 27b using type B specimens with a cylinder and cup fit for lateral restraint as shown in Figure 15. Lateral restraint provided by type B specimens is seen to permit the use of bonding pressures up to 10 ksi higher than those for type A specimens without making supporting tooling necessary in the temperature range studied.

From the above, it is seen to be possible to carry out diffusion welding operations for the RS/PM alloy at temperatures up to 600 °C (1112 °F) without having to provide tooling for lateral support to the materials being bonded. Conventional aluminum alloys can not be subjected to diffusion welding operations at temperatures above 500 °C (932 °F) without completely structurally supporting the alloy. This was verified by a bonding trial on a 2024-T6 aluminum alloy type A sample at 530 °C (986 °F). The sample crumbled on the application of temperature and a pressure of about 1 ksi normal to the faying surfaces. For the RS/PM alloy, however, higher temperature bonding processes such as transient liquid-phase bonding would be possible without having to support the material laterally.
The following sections describe the characteristics of welded specimens. First, Type A straight cylinder specimens are discussed. The welding parameters for these were chosen as above to result in base material softening of up to 10 VPN and a deformation of 5-10 % at the faying surfaces. Temperatures were varied from 350 to 500 °C (662 to 932 °F). The corresponding welding time and pressure were chosen from Figures 20 (curve A) and 31a respectively. Some samples were also bonded at conditions that resulted in a) no deformation at the faying surfaces and b) increased deformation at the faying surfaces. Only the final iteration of bonded specimens are covered as these are felt to adequately describe the diffusion welding characteristics of the alloy. The next section covers Type B specimens that were bonded. Parameters that yielded good strength values with Type A specimens were tried on Type B specimens to evaluate the effect of the lateral restraint offered by the cylinder-cup fit of these specimens. In addition, parameters chosen from the welding parameter zones for type B specimens were used. The final section covers transient liquid-phase bonded specimens.
5.6 Diffusion Welding Studies (Type A specimens)

Table 9 presents bonding parameters and strength values for diffusion-welded Type A Al-9Fe-4Ce specimens. Diffusion welding parameters were chosen initially for specimens D1 to D6 from the bonding parameter zones developed for the alloy. The welding temperatures were varied from 350 to 500 °C (662 to 932 °F), representing a variation of bonding temperature of nearly 75 °C (135 °F) from the extrusion temperature of the alloy. At each temperature, the time of bonding was chosen from curve A in Figure 18 and the welding pressure from Figure 31a. Specimens D7 to D9 were used to investigate the effect of lower pressures that resulted in no macroscopic deformation upon bonding. The effect of longer welding times and pressures that resulted in increased deformation was investigated using specimens D10 to D12.

Figures 28a and 30a to 34a show optical macrographs of longitudinal cross-sections of welded specimens D1, D3, D4, D6 (welded at different temperatures), D10 and D12 (bonded with parameters that resulted in increased deformation) respectively. The observed differences in the sizes of the specimens is discussed later in the section on mechanical testing. Figures 28b and 30b to 34b
show optical micrographs of the same bonds in the polished condition and Figures 28c and 30c to 34c the same sections etched with Keller's reagent.

5.6.1 Type A specimens bonded using developed parameter zones 5.6.1.1. Macro- and Microstructures

5.6.1.1.1 Low temperature regimes

Figures 28a to c show optical macro- and micro-graphs of a longitudinal cross-section of specimen D1 bonded at 350 °C (662 °F), about 75 °C (135 °F) below the extrusion temperature of the alloy. Figure 28a (Specimen D1 polished in Mastermet solution) shows a relatively uniform, silver-rich darker etching region at the center of the specimen. When polished in the Mastermet solution, the specimen exhibited dark streaks with a pitted appearance, with the streaks inclined to the bond-line at nearly 45 degrees. The streaks extended through silver-rich intermetallic layers at the bond and terminated in dimple shaped regions on the base metal indicated by arrows in the figure. The dimple shaped regions contained
Table 9. Welding parameters and strength values of diffusion welded type A Al-9Fe-4Ce samples

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Welding Conditions</th>
<th>Deformation</th>
<th>Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>No.</td>
<td>Temp- Pressure time at the faying surface (%)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>temperature (ksi) (minutes)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Base alloy</td>
<td>60.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D1</td>
<td>350 15.7 60 8</td>
<td></td>
<td>19.6</td>
</tr>
<tr>
<td>D2</td>
<td>350 15.8 60 9</td>
<td></td>
<td>18.5</td>
</tr>
<tr>
<td>D3</td>
<td>400 14.0 30 13</td>
<td></td>
<td>22.1</td>
</tr>
<tr>
<td>D4</td>
<td>450 9.0 20 14</td>
<td></td>
<td>11.5</td>
</tr>
<tr>
<td>D5</td>
<td>450 9.0 20 14</td>
<td></td>
<td>10.1</td>
</tr>
<tr>
<td>D6</td>
<td>500 6.8 10 10</td>
<td></td>
<td>BH*</td>
</tr>
<tr>
<td>D7</td>
<td>350 10.0 60 0</td>
<td></td>
<td>BH*</td>
</tr>
<tr>
<td>D8</td>
<td>450 5.6 60 0</td>
<td></td>
<td>BH*</td>
</tr>
<tr>
<td>D9</td>
<td>450 6.0 60 0</td>
<td></td>
<td>BH*</td>
</tr>
<tr>
<td>D10</td>
<td>500 5.6 60 18</td>
<td></td>
<td>26.5</td>
</tr>
<tr>
<td>D11</td>
<td>500 5.6 60 19</td>
<td></td>
<td>BPM**</td>
</tr>
<tr>
<td>D12</td>
<td>500 7.0 60 55</td>
<td></td>
<td>41.9</td>
</tr>
</tbody>
</table>

* Broke in handling ** Broke in parent metal upon hammering
concentric rings and appeared to have undergone plastic flow. It appears that the deformation of the specimen during bonding may have occurred largely in and around the silver rich area, which was also at a higher temperature than other regions of the specimen. The slip appears to have been accommodated at the dimple shaped regions and also accompanied by the precipitation of a phase or phases along the slip planes. The phase/phases appear to have reacted with the Mastermet polishing solution and eroded during the polishing operation leaving behind dark streaks seen in the polished microstructure. The streaks did not consist of pores, as they were not seen in samples polished in alumina. The lighter silver rich layer at the middle of the bond in Figure 28b consisted of two distinct layers when examined at higher magnification, an outer layer with a greyish appearance and and inner layer that was lighter. These layers were intermetallics growing into what was originally a silver layer at the interface. The center and edges of the specimen showed no observable microstructural differences.
Figure 28. Light micrographs of diffusion weld in Al-Fe-Ce type A specimen D1: A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
In the etched condition (Figure 28c), the specimen showed a dark-etching region adjacent to the silver-rich layer and an outer light-etching region. The darker region indicates the extent to which silver diffused into the Al-9Fe-4Ce base material and the lighter region represents base material coarsened during the bonding operation. The streaks seen when polished in Mastermet solution were not seen in the etched condition.

Figures 29a, b and c show scanning-electron micrographs of the same specimen (D1). When polished in alumina, (Figure 29a) the specimen exhibited Kirkendall porosity (indicated by arrows in the figure) on the base material on either side of and adjacent to the silver rich intermetallic layers at the bond. Later analysis including large differences observed in the diffusion rates of Ag into Al and Al into Ag and fractographs of tensile tested specimens will be used to conclude that the porosity observed is in fact Kirkendall porosity.

Figures 29b and c show the etched condition. EDAX analysis of points marked A, B, C and D in Figure 29b is
Figure 29 Scanning-electron micrographs of diffusion weld in type A Al-Fe-Ce specimen D1: 
A) polished in alumina; B and C) Keller's Etch.
Table 10. Standardless EDAX analysis of locations marked A, B, C and D in Figure 29b (Specimen D1).

<table>
<thead>
<tr>
<th>Element</th>
<th>Location A</th>
<th>Location B</th>
<th>Location C</th>
<th>Location D</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt% Atom%</td>
<td>Wt% Atom%</td>
<td>Wt% Atom%</td>
<td>Wt% Atom%</td>
</tr>
<tr>
<td>Al (K)</td>
<td>12.5 36.4</td>
<td>14.3 39.5</td>
<td>73.5 89.3</td>
<td>83.0 93.0</td>
</tr>
<tr>
<td>Ag (L)</td>
<td>87.4 63.4</td>
<td>82.8 57.3</td>
<td>15.0 4.5</td>
<td>2.4 0.7</td>
</tr>
<tr>
<td>Ce (L)</td>
<td>0.0 0.0</td>
<td>0.6 0.3</td>
<td>1.5 0.3</td>
<td>5.0 1.1</td>
</tr>
<tr>
<td>Fe (K)</td>
<td>0.1 0.2</td>
<td>1.3 1.7</td>
<td>9.7 5.7</td>
<td>9.7 5.2</td>
</tr>
<tr>
<td>Zn (K)</td>
<td>0.0 0.0</td>
<td>1.0 1.2</td>
<td>0.4 0.2</td>
<td>0.0 0.0</td>
</tr>
</tbody>
</table>
given in Table 10. Locations A and B represent intermetallic layers growing into what was originally silver. Two distinct intermetallic layers are observable in Figure 29b. Location A or the bond center had a composition close to the intermetallic compound Ag2Al. It was a binary intermetallic compound with very little Fe or Ce content. The outer, grey-etching layer indicated as location B was a complex, multi-component intermetallic compound containing small amounts of Fe and Ce. The base material region represented by location C was found to contain little more than 4 atom % silver, which is approximately equal to the solubility limit of silver in aluminum at the bonding temperature of 350 °C. Some porosity was observed in this region, but the dark streaks in Figure 32b at 45 degree orientation to the bond line are not seen in this heavily-etched sample.

Location D contained less than 1 atomic % silver. Microstructural features in this region were somewhat coarser than that of the unaffected base metal far from the bond region, as shown in Figure 29b. The region was consequently observed to etch lighter than the base material in Figure 32c.
Knoop hardness testing with a 25 gram load showed the Ag$_2$Al and the multi-component intermetallic layers to be softer than the base metal. The base metal showed a Knoop hardness of 83, the multi-component intermetallic layer showed a Knoop hardness of 52 and the Ag$_2$Al layer a Knoop hardness of 45.

Locations A and D were nearly at the same distance from the original Al-Ag interface. Location D contained less than 1 atom % silver, while Location A contained between 30 and 40 atom % aluminum. Thus, aluminum from the Al-Fe-Ce base material had diffused into the original silver layer at a rate much faster than the rate at which silver diffused into aluminum. It appeared that the complex and fine intermetallics coupled with the high solute contents in the Al-Fe-Ce base material may have acted as barriers to the outward diffusion of silver. Since the silver layer was originally an electro-deposited pure layer, there was no similar barrier for the diffusion of aluminum into silver.

The much faster rates observed of diffusion of aluminum into silver than those of silver into aluminum would very likely result in Kirkendall porosity in the Al-Fe-Ce base material adjacent to the weld, even at the
high weld pressures involved. Such Kirkendall porosity is seen in Figure 33a adjacent to the bond. The porosity also appeared to form along streaks in the base material parallel to the extrusion direction. This is probably due to microstructural differences within the base material arising from differences in particulate cooling rates and consequent differences in the diffusion rates of aluminum away from the base material into the silver layer.

The combined thickness of the two intermetallic layers in Figure 29a is approximately 0.0016 inch versus the total thickness of 0.0012 inch for the silver layers of the two samples bonded. The much higher extent of diffusion of aluminum into silver than silver into aluminum with porosity formation in the base material adjacent to the intermetallic layers explains this greater thickness.

5.6.1.1.2 Welding Temperatures close to the extrusion temperature

Microstructural features exhibited by Figures 30a to c and 31a to c (Specimens D3 and D4 bonded at 400 and 450°C (752 and 842°F)) were very similar to those in Figures 28a to c and described above for specimen D1 (bonded at 350
C (662 °F)) in Figures 28a to c. There were no observable differences in the microstructure from the specimen center to the specimen edges. The combined thickness of the two silver rich intermetallic layers in each of these figures is 0.0012 inch, which was the same as the starting total thickness. The slightly increased deformation of these two specimens during welding in the lateral direction may have offset the increase in thickness due to the diffusion of aluminum into the silver layer.

5.6.1.1.3 High temperature bonding regimes

Microstructural features exhibited by Figures 32a to c (Specimens D6 bonded at 500 °C (932 °F)) were very similar to those in Figures 28a to c and described above for specimen D1. There were no observable differences in the microstructure from the specimen center to the specimen edges. The combined thickness of the intermetallic layers is an intermediate 0.0014 inch. The deformation experienced by this specimen was between that of specimens D1 and D3. In Figure 32b (Specimen D6), the central intermetallic layer with a composition close to Ag2Al was only a thin streak along
Figure 30. Light micrographs diffusion weld in type A specimen D3: A) Keller's etch. 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
Figure 31. Light micrographs of diffusion welded type A specimen D4: A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
Figure 32. Light micrographs of diffusion welded type A specimen D6: A) Keller's etch, 2-3 X;
B) polished in Mastermet solution;
C) Keller's etch
the bond center. The outer multi-component intermetallic layer accounted for most of the intermetallic layer thickness, coupled with increased porosity in the base material adjacent to the intermetallic layers. However, at intermittent locations, such as those marked A in Figure 32b, porosity close to the bond appeared to act as a barrier to diffusion and the central layer extended outward up to the porosity.

5.6.1.1.4 Bonding regimes resulting in increased deformation

Figures 33a to c show optical macro- and micrographs of a longitudinal section of specimen D10 (Bonding conditions: 500 °C/60 minutes/5.6 ksi). The macrostructure is uniform from the center of the specimen to its edges. The specimen in the as-polished condition showed dark streaks at an angle to the bond line terminating in dimple shaped features. The light-etching central layer in Figure 33c designated as A is again an intermetallic layer with a composition close to Ag2Al and very low Fe and Ce contents. The longer bonding times of this specimen appear to have resulted in a complex multi-phase structure enveloping the Ag2Al type intermetallic layer.

Figures 34a, b and c show optical macro- and
micrographs of a longitudinal section of specimen D12 (500°C / 7 Ksi/ 60 minutes) bonded with increased deformation. The specimen extensive deformation during bonding is clearly seen in Figure 38a. Figure 35a shows the streaks in the specimen at a 45 degree angle to the faying surfaces described earlier. In the etched condition, the specimen showed a thin Ag₂Al type intermetallic layer at the center and a dark-etching silver containing layer. The microstructure and the compositions are more clearly seen in the scanning-electron micrographs of the specimen described below.

Figures 35a, b and c show scanning electron micrographs of the bond region in specimen D12. When polished in alumina (Figure 35a) again shows Kirkendall porosity in the base material adjacent to the bond (indicated by arrows in the figure). Figures 35b and c are of the specimen in the etched condition*. Figure 35b shows an Ag₂Al type layer at some locations along the length of the bond center (marked A in the figure) enveloped by a multi-phase region. At other locations, small differences in the thickness of the original silver as plated and heterogeneous deformation appear to have
Figure 33. Light micrographs of a longitudinal diffusion welded type A specimen D10 bonded at 500 °C (932 °F)/60 minutes/5.6 ksi pressure:

A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
Figure 34. Light micrographs of diffusion welded type A specimen D12 bonded at 500°C (932°F)/60 minutes/7 ksi pressure: A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
Figure 35. Scanning-electron micrographs of diffusion welded type A specimen D12: A) polished in alumina;
Figure 35. Scanning-electron micrographs of diffusion (Contd.) welded type A specimen D12; B and C) Keller's Etch.
resulted in the absence of the Ag₂Al type layer in between the multiphase layers. Immediately outside the multi-phase region in the base material, Kirkendall porosity is seen on either side. The microstructure is very similar to that of specimen D10, except that the central Ag₂Al type layer is much thinner in specimen D12 due to its increased deformation during bonding.

5.6.2 Tensile strengths of the bonds in Type A specimens

Specimens D1 to D3 bonded at 350 and 400 °C, which are less than the hot extrusion temperature of the alloy, exhibited bond strengths greater than specimens D4 to D6, bonded at temperatures higher than the hot extrusion temperature of the alloy. Bond-strengths appeared to be limited by porosity and the effect of the porosity appeared to be more severe in specimens bonded for short times above the extrusion temperature of the alloy.

Specimen D10, which showed plastic deformation, exhibited higher strength than specimens D1, D3, D4 or D6. The increased deformation appears to have promoted metallic contact (local welding) and improved bond strength. Specimen D12, which exhibited much higher deformation showed a significantly improved bond-strength.
Specimens D7, D8 and D9 (bonded at temperatures from 350 to 450°C (662 to 842°F)), which did not deform macroscopically during bonding, came apart in handling. The silver-rich intermetallic layer peeled off on either side probably due to separation by Kirkendall porosity. Evidence of bonding was observed at the central regions of the specimens. Though these specimens did not exhibit observable macroscopic deformation, some interfacial extension appeared to have occurred at the central regions resulting in bonding. The outer edges of the specimen had not been bonded. Increased bonding times at these lower pressure/no deformation bonding regimes did not improve bond strengths. Hence some deformation at the faying surface appears necessary in view of the vastly different rates of diffusion observed.

5.6.3 Hardness profiles across bonds in type A specimens

Figure 36 shows microhardness profiles across bonds in specimens D1, D3, D4 and D6 and Figure 37 across specimens D10 and D12. The hardness drop at any location in specimens D1, D3 and D4 does not exceed 10 VPN. The bonding time-temperature combinations for these specimens had been chosen from curve A of Figure 18 and were expected to result in a hardness drop not exceeding 10 VPN in the base material upon bonding. The methodology of
Figure 36. Microhardness profiles across diffusion welded type A Al-9Fe-4Ce alloy specimens D1, D3, D4 and D6. Distances are from the interface between the base material and the intermetallic layers into the base material.
Figure 37. Microhardness profiles across diffusion welded type A Al-9Fe-4Ce alloy specimens D10 and D12. Distances are from the interface between the base material and the intermetallic layers into the base material.
developing bonding time-temperature parameter zones for the alloy using coarsening studies to limit softening during bonding to any desired value was successful. Specimen D6 shows a somewhat higher softening than expected. The bonding temperature of 500 °C (932 °F) used for this specimen was relatively high with respect to the melting point of aluminum. Hence, any small variations on bonding time may have resulted in hardness differences. The secondary hardening at this temperature seen in Figure 18 would also be sensitive to any variations in bond time. Even in this case, the scatter in the hardness is within 10 VPN from the predicted value. Specimens D10 and D12 show much higher softening as expected due to the increased bonding time at 500 °C (932 °F).

5.6.4 Fractography of Al-9Fe-4Ce base material and type A diffusion welded specimens

Figures 38a and b show scanning-electron fractographs of a Al-9Fe-4Ce base material sample that was bent to fracture. The material exhibited a laminated fracture surface and also some texture along the extrusion direction. The fine grained microstructure of the base material is evident in Figure 38b.
Figure 38. Scanning-electron fractographs of as-received Al-9Fe-4Ce alloy bent to fracture.
Figures 39a, b, c and d show scanning-electron fractographs of the tensile test coupon of specimen D1 bonded at 350°C (662 °F). The light region in Figure 43a on the left marked A indicates the intermetallic layer seen at the bond center in Figure 29 and the darker region on the right marked B indicates the base material with small amounts of silver dissolved in it. Dark spotty regions in the base material were Kirkendall pores, which had been flattened due to the deformation of the specimen during bonding. The porosity does not appear in the region marked A. Figure 39b shows the porosity to have formed preferentially along streaks in the base material aligned along the extrusion direction. This preferential formation of porosity was also observed in Figure 29b, a scanning-electron micrograph of a section of the same specimen. Figure 39c shows one of the dark spots in region B of Figure 39a at a higher magnification. Porosity (marked by an arrow) is observed at the center of the dark spot. Figure 39d shows the region marked A in Figure 39a at a higher magnification. No porosity is seen in this figure, which shows the intermetallic side of the fracture.

The interfaces between the base material on either side and the intermetallic layers appear to have given
way initially due to the Kirkendall porosity, ultimately leaving the layer alone to support the load. The intermetallic layer then fractured leaving parts of it behind on both sides of the fractured joint. This was evidenced by the cracks running through the intermetallic layers in Figure 39a.

Figures 40a, b and c show fractographs of specimen D3 bonded at 400 C (752 F). The fractured surface is very similar to that in Figure 39, except that the Kirkendall porosity was somewhat finer and more scattered. This was probably due to the slightly higher deformation of this sample during bonding. Figure 43b shows one of the dark spots at a higher magnification and Figure 39c the intermetallic layer side of the fracture at a higher magnification. Once again, it is seen that porosity was observed only on the base material side of their fracture and not on the intermetallic compound as expected from observed diffusion rates. The less extensive porosity in this specimen also resulted in this sample exhibiting an improved bond strength over specimen D1.

Figures 41a and b show scanning-electron fractographs of the tensile-test coupons of specimen D12 which exhibited increased deformation upon bonding. Figure 35a, a longitudinal section of this specimen had shown an
intermittent Ag₂Al type layer along the length of the bond at the bond center enveloped by a multi-phase region. At other locations deformation of the specimen had promoted contact between the multi-phase regions without the Ag₂Al type layer in between. The fracture surface reflects such a morphology with the silver rich layer seen intermittently.

Figures 42a and b show scanning-electron fractographs of the central and outer regions of the same specimen. The central region exhibited finer microstructural features than the outer regions. As mentioned in section 2.2 (Ref.24), the central regions of the faying surfaces experience a much higher interfacial extension than the outer regions of the sample. The silver and later the intermetallic layers upon recrystallization would then be expected to show finer microstructural features at the central regions than at the outer regions. Such differences, however, could not be observed in the complex microstructure of the longitudinal sections of the bond discussed earlier.

5.7 Diffusion Welding Studies (Type B specimens)

Table 13 presents welding parameters and strengths of diffusion-welded Type B Al-9Fe-4Ce specimens. It is shown in Figures 27a and b that for identical bonding time and
Figure 39. Scanning-electron fractographs of diffusion welded type A specimen D1: A) Region A (base material with dissolved silver) showing Kirkendall porosity and region B (intermetallic layer); B) Kirkendall porosity is formed along streaks in the base material aligned in the extrusion direction.
Figure 39. Scanning-electron fractographs of diffusion (contd.) welded type A specimen D1: C) A Kirkendall pore (shown by an arrow) in region B of Figure 39a; D) intermetallic layer in region A of Figure 39a.
Figure 40. Scanning-electron fractographs of diffusion welded type A specimen D3: A) Regions A and B similar to those in Figure 39a; B) Kirkendall pore in region A of Figure 40a; C) intermetallic layer in region B
Figure 41. Scanning-electron fractographs of diffusion welded type A specimen D12: A and B) fracture surfaces showing the discontinuous $\text{Ag}_2\text{Al}$ type intermetallic layer observed at the bond.
Figure 42. Scanning-electron fractographs of Specimen D12: A) specimen center; B) specimen edges.
Table 12. Welding parameters and strength values of diffusion-welded Type B Al-9Fe-4Ce samples

<table>
<thead>
<tr>
<th>No.</th>
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<th>Temp-Pressure</th>
<th>time</th>
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</table>
temperatures, higher welding pressures could be applied on Type B specimens than on Type A specimens without increasing the amount of macroscopic deformation at the faying surfaces. Alternatively, bonding times could be increased for the Type B specimens, while maintaining the same welding temperature and pressure. Thirdly, deformation of the specimen at the faying surfaces may be reduced for the same welding temperature, time and pressure. The purpose of this section was to study the effect of increased welding pressures and/or times on weld quality. Specimens D24, D25 and D26 were of Type B2. These were fabricated with a cup and cylinder fit as shown in Figure 11 with a 5 percent diametral clearance between the cylinder and the cup. These specimens were intended to restrict the deformation at the faying surfaces to about 5%.

5.7.1 Macro- and microstructures of the welds

Figures 43a, b and c show optical macro- and micrographs of specimen D15 (Bonding conditions: 500 °C (932 °F)/30 minutes/8 ksi pressure). From Figure 43a, the cup-cylinder fit of the type B specimens is seen to restrain the sample from deforming at the faying surfaces, even though considerable deformation is observed away from the faying surfaces at locations that
Figure 43. Light micrographs of diffusion welded type B specimen D15 (bonded at 500°C/30 minutes/8 ksi pressure: A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
Figure 43. D) Scanning-electron micrograph of diffusion welded type B specimen D15.
experience even lower temperatures than at the faying surfaces. Figure 43b shows dark streaks at the bond area terminating in dimpled-shaped features on the base material similar to those seen earlier in Figures 28 to 33 for Type A specimens. The difference is that these streaks are not inclined at 45 degrees to the weld line, but rather are oriented at right angles to them. The lateral restraint at the faying surfaces due to the cup-cylinder fit of these specimens imposed considerable shear stresses in the direction of the interface. Hence maximum shear stress may not have been at 45 degrees to the bond line in these specimens. The formation of these streaks thus is related to the shear stresses and deformation experienced during bonding. Figure 43d shows a scanning-electron micrograph of a longitudinal section of specimen D15. EDAX analysis of the bond region yielded results very similar to those in Table 10 for Specimen D1. The central layer was a binary Ag$_2$Al type intermetallic layer with very little Fe, Ce or Zn. The microstructure and the composition are thus very similar to those described previously for sample D10.

Figure 44a and b show optical macro- and micrographs of specimen D19, which was bonded for 2 hours at 400 °C (752 F). The macrograph shows no macroscopic deformation.
Figure 44. Light micrographs of diffusion welded type B specimen D19 (bonded at 450°C (842°F)/120 minutes/7 ksi pressure): A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's Etch.
Figure 45. Light micrographs of diffusion welded type B specimen D13 (bonded at 500 °C/60 minutes/5.6 ksi pressure): A) Keller's etch, 2-3 X; B) polished in Mastermet solution; C) Keller's etch.
Figure 44b shows a good Ag-Ag bond, but the bond failed at the interface between the base material and the silver rich intermetallic layers. Kirkendall porosity and the high shear stresses along the joint during the bonding operation and subsequent cooling appear to have caused the separation from the base material. Figure 45a,b and c show optical micrographs of a longitudinal section of specimen D13, which was bonded using the same bonding conditions that had been used for type A specimen D10 (500°C (932°F)/60 minutes/5.6 ksi pressure). While specimen D12 had deformed 18 percent as measured by its increase in surface area at the faying surfaces and had shown a joint tensile strength of 26.5 ksi, D13 did not deform macroscopically and came apart in handling. The silver-silver bond at the interface was satisfactory, but the silver rich intermetallic layer peeled off on either side.

5.7.2 Tensile strength of the bonds in type B specimens

In general, strengths of bonds made with type B specimens were poor, as shown in Table 12. Bonding conditions that yielded joint strengths greater than the tensile strength of silver with type A specimens resulted
in bonds that came apart in handling. Limitation of deformation at the faying surfaces by this specimen configuration appears to have adversely affected joint strengths by permitting Kirkendall porosity to separate the base material from the silver rich layer. The increased pressures permitted by the type B specimen configuration did not suppress the formation of or heal porosity.

Type B2 specimens, in general resulted in poor joint strengths. The fixturing available could not align the mating surfaces concentically. Hence deformations at the faying surfaces were less than the expected 5% increase in area. These were not sufficient to overcome the effects of Kirkendall porosity in these joints.

5.7.3 Hardness profiles across bonds in type B specimens
Figure 46 shows microhardness profiles across the bond in specimens D15 and D22. The profiles in general are similar to those of type A specimens bonded under the same conditions.

5.7.4 Fractography of type B diffusion bonded specimens
Figure 47 shows a scanning-electron fractograph of the type B specimen D13. The fractured surface was decorated with extensive Kirkendall porosity, which had virtually
separated the silver rich intermetallic layer from the base material. Figure 48 shows a fractograph of the tensile test coupon of specimen D15. The increased deformation of this sample resulted in a reduction in the extent of Kirkendall porosity at the fractured surface and improved bond strength.

In summary, Type B specimens were found to be unsuitable for bonding this alloy. Their effect in limiting deformation at the faying surfaces and permitting increased bonding pressures or times was not beneficial in improving joint strengths.
Figure 46. Microhardness profiles across diffusion-welded type B Al-9Fe-4Ce alloy specimens D15 and D22. Distances are from the interface between the intermetallic layers and the base material into the base material.
Figure 47. Scanning-electron fractograph of diffusion-welded type B Specimen D13.
Figure 48. Scanning-electron fractographs of diffusion welded type B Specimen D15: A) fracture surface at low magnification; B) Kirkendall porosity in the base material; C) intermetallic layer with no porosity.
5.8 Transient liquid-phase bonding of Al-9Fe-4Ce alloy

Table 12 gives bonding parameters and strength values of transient liquid-phase bonded Al-9Fe-4Ce alloy samples. Specimens TLP 1, 2 and 3 bonded at temperatures ranging from 530 to 565°C (986 to 1049°F) came apart in handling upon cooling from the bonding temperatures. Visual examination of the faying surfaces showed no evidence of melting or the formation of a eutectic liquid phase. The formation of eutectic liquid was not observed upon visual examination even in sample TLP 3, which had undergone some deformation at 565°C (1049°F). Consequently higher temperatures ranging from 575°C to 600°C (1067 to 1112°F) were used in subsequent bonding trials.

5.8.1 Macro- and Micro-structures of transient liquid phase bonded Al-9Fe-4Ce specimens

Figures 49a, b and c show optical macro- and micrographs of a longitudinal section of transient liquid-phase bonded sample TLP9 bonded at 575°C/2.5 minutes + 400°C/40 minutes. The specimen was bonded using one electroplated and one non-plated mating parts. It is seen to have undergone considerable plastic deformation during the bonding operation. Figure 28b shows dark streaks
Table 12. Bonding parameters and strength values of transient-liquid phase bonded Al-9Fe-4Ce samples

<table>
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Figure 49. Light micro-graphs of transient liquid phase bonded specimen TLP 9 (bonded at 575 °C/2.5 minutes +400 °C/40 minutes): A) Keller's etch; B) polished in Mastermet solution; C) Keller's etch.
Figure 50. Scanning electron micrographs of transient liquid phase specimen TLP-9, Keller's etch.
around the bond terminating in dimple shaped features as in diffusion welded samples shown in Figures 28 to 33. The streaks are not perpendicular to the bond line as in diffusion welded type B specimens but inclined at 45 degrees to it. The bonding temperatures for these specimens were rather high and the female mating part of the specimen is seen to deform with the male mating part rather than constrain it from deforming. Consequently, shear stresses due to the lateral constraint that the specimen experiences were not very high and the dark streaks formed at 45 degree angles. Figure 28c shows a dark etched, silver rich thin layer at the bond-line marked A in the figure. Adjacent to this dark layer on one side of it a lighter region designated B and a light etching layer designated C were observed. On the other side of layer A, a dark region similar to B was not observed, but only a lighter etching region similar to C was seen. These regions can be seen more clearly in scanning-electron micrographs of the section shown in Figures 50 a and b. However, in the scanning-electron micrographs, silver rich zones appear brighter than non-silver rich areas. Both the micrographs in Figure 50 show that silver was present predominantly on only one side of the bond. If eutectic liquid had formed when bonding temperature was 575 °C (1067 °F) and wetted both mating
pieces, such differences in silver content on the two sides of the bond would not have existed. Hence, the bond was essentially a deformation bond. The little silver seen in the mating part that was not originally silver plated must have diffused into it after deformation during bonding created nascent oxide-free surfaces in contact with silver. Another conclusion that can be drawn from Figures 49 and 50 is that even the temperature of 575 °C (1067 °F) was not sufficient to cause the formation of eutectic liquid. For, if eutectic liquid had formed, it would have been expected to wet one side of the bond as well as the other.

Figures 51a, b and c show optical macro- and micrographs of a longitudinal section of transient liquid phase bonded specimen TLP 11 bonded at 590 °C (1094 °F)/30 minutes. This specimen had been bonded using mating parts that had both been silver-plated. The specimen did not experience any macroscopic deformation during bonding, as seen from Figure 51a. Figure 51b again shows dark streaks around the faying surfaces, similar to the ones seen earlier. These streaks were not seen in heavily etched sections, such as Figure 51c. A clear gap is seen between the two mating parts in Figures 51b and c.
Figures 52a, b, c and o show scanning-electron micrographs of the specimen TLP-11. Most of the length of the bond exhibited a structure similar to that shown in Figure 52a. EDAX analysis very close to the faying surfaces on either side showed an Ag content of less than 1 atom %. A few isolated areas were found along the bond with structures similar to that in Figure 50b. Silver containing areas with a lighter appearance are seen on both sides of the bond. A silver containing area was also found at the corner of the sample marked by a small arrow in Figure 51a. EDAX analysis showed this area to have an Ag content of nearly 5 atom %. The other corner of the sample did not exhibit any such silver containing areas. It appeared that while Ag-Al eutectic liquid did form in this sample, it could not wet the surface and tended to flow to a few discreet locations. Such wettability problems had also been experienced by Cline (Ref.27) in the transient liquid-phase bonding of aluminum using electro-plated silver.

5.8.2 Tensile strengths of transient liquid-phase bonded joints in the Al-9Fe-4Ce alloy.

The tensile strengths of specimens TLP-9 and TLP-10 bonded under the same conditions (575 C/2.5 minutes + 400
Figure 51. Light micro-graphs of transient liquid phase bonded specimen TLP-11: A) Keller's etch; B) polished in Mastermet solution; C) Keller's etch.
Figure 52. Scanning electron micrographs of transient liquid-phase bonded specimen TLP-11, Keller's etch.
C/40 minutes) were nearly equal, showing repeatability of results in this study. It has been shown possible to obtain joint strengths of 10-20 ksi using this method of bonding, but the joints appeared to be diffusion/deformation welds rather than transient liquid-phase bonds.

5.8.3 Microhardness profiles across TLP bonds

Microhardness profiles across some of the bonds listed in Table 12 are shown in Figure 53. The softening of the base material was somewhat greater than in diffusion welded samples joined using the diffusion welding parameter zones developed.

5.8.4 Fractography of TLP bonds

Figure 54 shows a scanning-electron fractograph of the tensile coupon fracture surface in specimen TLP 11 (590 °C (1094 °F)/30 minutes-no deformation). The failure of the eutectic-liquid to wet the faying surfaces is evident from the fractograph. Intermittent, bright silver-rich particles were seen (marked by small arrows in the figure) on the fracture surface rather a uniform distribution of silver.

Figure 55 shows scanning-electron fractographs of the fracture surfaces of the tensile test coupon of specimen
TLP-9. Figure 55a shows many bright silver containing areas, while Figure 55b does not show such areas.

In summary, Al-Ag eutectic appeared to form only at temperatures of 590 °C (1094 °F) or above. At a temperature of 575 °C (1067 °F), the liquid did not form. Non-wetting of the faying surfaces by the eutectic liquid was found result in poor joint strengths. The joints that exhibited strengths of 10-20 ksi were essentially deformation/diffusion welds.
Figure 53. Microhardness profiles across transient liquid-phase bonded Al-9Fe-4Ce alloy specimen TLP 9.
Figure 54. Scanning electron fractograph of tensile test specimen TLP-11 (590 °C (1094 °F) / 30 minutes).
Figure 55. Scanning electron fractographs of tensile test specimen TLP-9 (575 °C (1067 °F)/2.5 minutes + 400 °C (752 °F)/40 minutes).
6. Summary of Results and Discussion

6.1 Diffusion Bonding Parameters

The methodology for arriving at optimum diffusion welding parameters for the RS/PM Al-Fe-Ce alloy differed from that employed for conventional aluminum alloys. This was because excessive coarsening of the alloy microstructure during the bonding operation would adversely affect its high temperature serviceability. A method had to be found to quantify the coarsening characteristics of the alloy at different welding time-temperature-pressure combinations and use the same to arrive at optimum welding parameters. Since the alloy is strengthened by dispersoids that do not dissolve in it in the solid state, higher temperature bonding regimes were likely to be attractive. The methodology developed for arriving at bonding parameters had to consider the high temperature bonding characteristics of the alloy as well.

The development of bonding parameter zones described in Figures 20 and 31 represents a systematic approach to the selection of diffusion welding time, temperature and pressure combinations for the RS/PM alloy. At any
selected welding temperature, the time of welding that limited softening of the base material to a desired value and the welding pressure that resulted in 5-10 % deformation at the faying surfaces could be readily chosen. Welding temperatures for this dispersion strengthened alloy could be much higher than 200 °C (392 F), the approximate recrystallization temperature of the silver interlayer. Based on previous studies, the time of welding chosen by the above approach should consequently be adequate for the formation of a satisfactory silver-silver bond at the interface. The bonding pressure so chosen appears to be appropriate, since welding pressures reported in literature for aluminum alloys slightly exceed their respective compressive yield strengths at the bonding temperatures. This methodology for the determination of optimum diffusion welding parameters may be generally applied to any RS/PM alloy.

The above approach was also found to be successful in limiting the decrease in the hardness of the base alloy during welding. From Figure 40, the hardness decrease in specimens welded at temperatures of 350 to 500 °C (662 to 932°F) with other parameters chosen from Figures 20 and 31a was found to be 10 VPN or less, as predicted by the parameter zones developed.
A significant difference between the welding parameter zones for the RS/PM alloy and those of conventional aluminum alloys is that at temperatures up to 600 °C (1112 °F), it is possible to carry out diffusion welding operations without having to provide tooling for lateral support to the materials being bonded. The conventional aluminum alloys can not be subjected to diffusion bonding operations at temperatures above 500 °C (932 °F) without completely structurally supporting the alloy. This was verified by a bonding trial on a 2024-T6 aluminum alloy type A sample at 530 °C (986 °F). In addition, the conventional alloy would need post-heat treatment to restore mechanical properties degraded by the bonding operation. If the particular conventional alloy is not heat-treatable, there is no way to retain/regain its mechanical properties. For this reason, one would expect any diffusion welding operations above 500 °C (932 °F) to be unattractive for conventional aluminum alloys. This would include transient liquid-phase bonding using a silver-aluminum eutectic liquid to form the bond. For the RS/PM alloy, however, such transient liquid-phase bonding would be theoretically possible without having to support the material laterally.
Investigation of diffusion welding of the Al-Fe-Ce alloy in this study did involve some macroscopic deformation at the faying surfaces. Consequently, the welds prepared were deformation/diffusion welds and not strictly diffusion welds in the strict sense of the term (ref. 13).

6.2 Cleaning and Electro-plating Procedures

In specimens D1 to D6, which were diffusion welded at temperatures from 350 to 500 °C (662 to 932 °F), the distribution of aluminum at the bond center was uniform along the length of the bond. 30-40 atomic % aluminum was present at the bond center even for the relatively short bonding times employed in the work. This shows that there were initially no barriers to the diffusion of aluminum into the silver layer. If the cleaning procedures were not satisfactory or if the electro-plate adhesion was not satisfactory, such uniform diffusion would not have been possible. The cleaning, zincating and electro-plating procedures employed for the alloy and described in Tables 5 and 6 hence appeared to be satisfactory.
Calculation of interdiffusion coefficients and prediction of growth rates of intermetallic layers was found to be not feasible for the RS/PM alloy due to the complexity of its microstructure and its multi-component chemistry. It was noted from the background section that Albright (Ref. 32) had chosen a steel with a very low oxygen content for his work on deformation welding and interdiffusion studies. This was because high oxygen contents in the steel were known to significantly limit interdiffusion with aluminum. Shewmon (Ref. 17) found that differences in the silicon contents of two steel specimens made into a diffusion couple caused uphill diffusion of carbon to occur. The Al-Fe-Ce alloy used in this study a complex multi-component chemistry with impurity elements like oxygen and hydrogen also being picked up during the processing of the alloy. The effect of such complex chemical composition and microstructure on interdiffusion with silver are difficult to predict and would need a separate detailed study.
Bond strengths of all diffusion welded specimens was less than 50% of starting base metal strength. The only exception was when a type A straight cylinder specimen was deformed more than 50%. The resulting bond strength was about 70% of base metal strength. Hence clearly the approach of diffusion welding did not result in high strength bonds. In cases where the specimen did not experience any macroscopic deformation during bonding it came apart upon cooling and handling. Diffusional porosity was clearly identified to be the reason for generally poor joint efficiencies. The observations that support this conclusion were many and are summarized below:

1) Highly unequal diffusion rates of Al into Ag and Ag into Al were observed.

2) Porosity was observed only on the base material side of welded specimens (the faster diffusing species) in the polished condition and not on the side of the intermetallics. This porosity was also found to be aligned along streaks in the microstructure of the base material oriented parallel to the extrusion direction. The streaks could not be identified as having formed from Zone A or zone B solidified powder.
microstructures.

3) Porosity was observed only on the base material side of fracture surfaces. Porosity again was found to be preferentially aligned.

4) The intermetallic compounds formed at the bond during the bonding operation were found to be softer than the base material. This agrees with the work of McEwan and Milner (Ref.33) who found that silver-aluminum intermetallics did not embrittle diffusion bonds even after long exposures to high temperatures, which resulted in the growth of their thickness. Thus, if a way can be found to limit porosity adjacent to the bond, the joints may become satisfactory. This may attempted by using hot isostatic pressing as the method of diffusion bonding. If the intermetallic layers can be made to be thick, they may also act as diffusion barriers during service and not allow porosity to develop. This aspect appears to be worth studying as a future effort.

Earlier work on diffusion welding of aluminum alloys did not report strengths being limited by Kirkendall porosity. In fact McEwan and Milner (Ref.33) found that silver-aluminum bonds that were heat-treated at 540°C
(1004°F) for 60 minutes did not show any reduction in bond strength due to post-heat treatment. Dini (Ref.48), who reviewed the use of silver as a diffusion aid in a variety of materials, did not encounter any diffusional porosity in aluminum alloys diffusion-bonded with silver interlayers. Hence, the generation of Kirkendall porosity in the diffusion welds in the RS/PM alloy appear to be due to the nature of its alloying elements and its complex microstructure. However, very little diffusion data is available for this class of alloys.

6.5 Transient-liquid Phase Bonds

The eutectic reaction between silver and aluminum in the RS/PM Al-9Fe-4Ce base material was found to occur at a temperature above 575°C (1067°F). This was evidenced by the grossly unequal distribution of silver across the bond in Specimen TLP 9 bonded at 575°C and soaked at 400 C (842°F). Non-wetting of the base material by the eutectic-liquid formed with the electro-plated silver layer was shown to prevent the formation of a satisfactory eutectic-bond in this study.
7. CONCLUSIONS

1. Short term coarsening studies were successful in establishing time-temperature regimes for diffusion welding an Al-9Fe-4Ce alloy that limited softening of the base material during the welding operation.

2. Pressure-temperature zones in which the alloy can be diffusion welded with and without structural support were experimentally determined. At temperatures up to 600°C (1112°F), it was possible to weld the alloy without structural support.

3. Diffusion welding operations were carried out using time-temperature and pressure-temperature zones established as above with silver interlayers. Joint strengths increased with increasing specimen deformation during welding. Specimens that experienced no deformation during the diffusion welding process, as ideally they should not as per the definition of the process, came apart upon cooling and handling. Kirkendall porosity formed at the interface between intermetallic layers formed during bonding and the base alloy were found to embrittle the welds. As a consequence, most of the welds
carried out in this study involved some macroscopic deformation at the faying surfaces. Joint efficiencies of nearly 70% were achieved in specimens that experienced about 50% increase in area at the faying surfaces during welding.

4. The complex microstructure and chemical composition of the Al-9Fe-4Ce alloy severely restricted the diffusion of silver into the base material. Aluminum from the base material however, diffused freely into the silver layer, resulting in the formation of Kirkendall porosity. Joint strengths were limited by Kirkendall porosity rather than the nature of microstructure that developed during bonding.

5. Cup-cylinder specimen geometry was shown to provide lateral restraint from deformation at the faying surfaces, which permitted the use of increased welding pressures. But the increased welding pressures did not aid in minimizing or healing the porosity.

6. The temperature-pressure regimes of diffusion welding for this alloy indicated that transient liquid-state bonding using a silver-aluminum eutectic may be more attractive for this class of aluminum alloys than for
conventional aluminum alloys. This is because at temperatures up to 600°C (1112°F), the Al-Fe-Ce alloy can be theoretically diffusion welded without structural support. However, non-wetting of the base material by Al-Ag eutectic liquid formed using a silver electroplated interlayer was found to result in TLP bonds of poor quality.
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