JOURNAL OF MATERIALS SCIENCE 22 (1987) 115–122

Interaction of Al–Si, Al–Ge, and Zn–Al eutectic alloys with SiC/Al discontinuously reinforced metal matrix composites

W. C. MOSHIER, J. S. AHEARN, D. C. COOKE
Martin Marietta Corporation, Martin Marietta Laboratories, 1450 South Rolling Road, Baltimore, Maryland 21227, USA

Interactions between Al–Si, Al–Ge, and Zn–Al eutectic alloys with SiC whisker-reinforced aluminium metal matrix composites were studied as a function of temperature above the eutectic melting temperature. Penetration extended several millimetres into the composite for the Al–Si and Al–Ge alloys but was restricted to a thin surface layer (50 μm) for the Zn–Al alloy. The extent of the penetration zone for the aluminium alloys containing silicon and germanium was also affected by the thermal-mechanical treatment of the composite: limited penetration was observed for hot-pressed material whereas extensive penetration was observed for mechanically worked material. Mechanisms for the observed phenomena are discussed in terms of the wettability of the SiC whiskers by the eutectic alloys, the formation of channels during mechanical working as well as the fine grain size of the composite.

1. Introduction

Recent interest in using silicon carbide/aluminium (SiC/Al) discontinuously reinforced metal matrix composites in actual prototype structures has generated a need to investigate methods to reliably join this family of materials. In past work on this material, fusion welding methods using gas metal arc or gas tungsten arc welding processes [1–3] to join SiC to itself and to wrought aluminium alloys were examined. Recently inertia welding, joining SiC/Al to wrought aluminium [4, 5] and SiC/Al to itself [5], has been used with excellent joint efficiencies.

We recently examined the brazeability of SiC/Al composites having SiC whisker (SiC_w) or SiC particle (SiC_p) reinforcement [6]. A discussion of the brazing methods and the resulting joint strengths is presented [6]. This paper presents our observations of composite interactions with eutectic alloys commonly used in brazing, as well as other eutectic alloys, with the aim to understand the dominant mechanism(s) of interaction as they occur in these materials.

Although there is ample literature discussing the interaction of braze alloys and wrought aluminium alloys (see, for example, [7]), there is very little work on the brazeability of discontinously reinforced aluminium metal matrix composites. We therefore took a fundamental approach in examining the interactions that occur between SiC/Al composites and the eutectic alloys conventionally used for brazing aluminium. The conditions of the composite, whether it was in the hot-pressed condition, extruded, hot isostatically pressed (HIPped), or heat treated, was varied to help elucidate the mechanism of eutectic alloy interaction. In addition, wetting tests were conducted at specific temperatures and the microstructure of the composite subsequently examined. This work had led us to better understanding of eutectic alloy interactions with SiC/Al composites as discussed in Section 4.

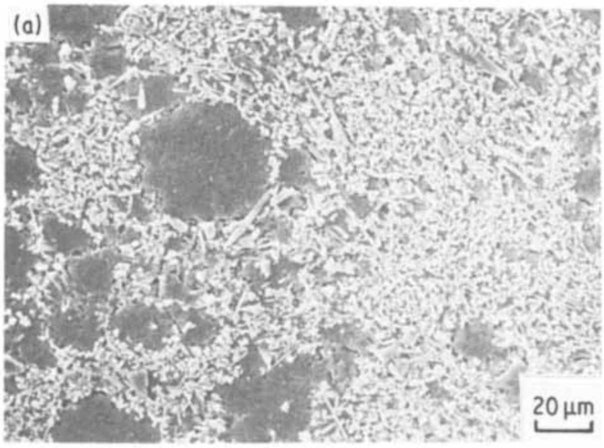
2. Experimental procedure

The metal matrix composites contained either 20 or 25 vol % SiC_w in a 6061 Al matrix (ARCO Chemical, Silag Operation, Greer, South Carolina) or 20 vol % SiC_p (DWA Composite Specialties, Chatsworth, California) in the same matrix; however, the work described below will focus on the interaction of eutectic alloys on the whisker-reinforced material. Several different forms of material were used during this study: (1) cold compacted and hot-pressed (no mechanical work), (2) plate form (extended from hot-pressed billet and then cross-rolled, and (3) plate which had been HIPped at 30 000 psi (210 N mm^{-2}) and 500° C for 2 h. All of the material had to be degassed prior to analysis to remove residual gas in the composite [1]. The interaction of the aluminium composite with binary eutectic mixtures of aluminium containing either silicon, germanium, or zinc was studied. The chemical compositions and melting temperature of each alloy are listed in Table I. The interaction of the eutectic alloy with the composite was studied by heating the materials in an evacuated (1 mPa) quartz tube. The materials were prepared for the vacuum wetting experiments by mechanically polishing each surface with 600-grit SiC paper, and then applying a thin coating of electroless nickel which acted to break

TABLE I Binary eutectic alloys

Alloy	Al composition (wt %)	Melting temp. (° C)
Al–Si	87.4	577
Al–Ge	47.0	424
Zn–Al	5.0	382

0022–2461/87 $03.00 + .12



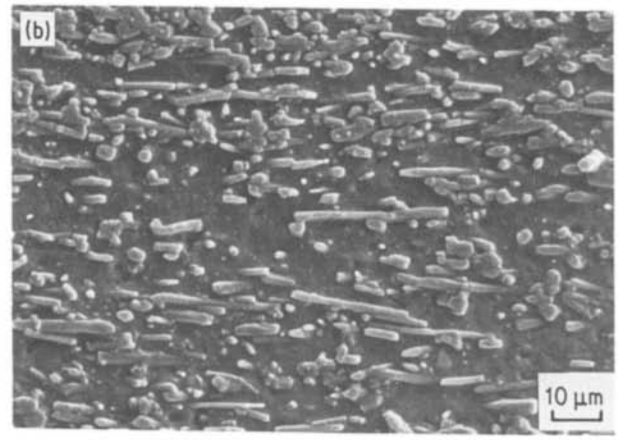


Figure 1 Microstructrual features of SiC_w/6061 Al in the: (a) hot-pressed billet before fabrication of plates, and (b) after extrusion and cross-rolling to make 3.2 mm (0.125 in.) thick plates.

down the surface oxide during brazing [8]. After the eutectic alloy melted and wet the composite, the sample was allowed to cool under vacuum. In our tests, each sample was held at the test temperature for less than 30 sec.

Following the vacuum wetting test, the samples were cross-sectioned, polished, and etched. A modified Keller's reagent was used to reveal the structure of the region affected by the eutectic alloy. Samples were prepared for scanning electron microscopy (SEM) by coating them with a thin conductive layer of platinum to reduce charging on the SiC. Thin foils prepared for transmission electron microscopy (TEM) were taken from wafers of the composite that had been mechanically polished to a thickness of 25 μm and milled using argon ion sputtering to provide electron-transparent foils.

3. Results

3.1. Microstructure

Examination of the composite revealed a strong influence of the processing history on the microstructure of the material. The hot-pressed billet microstructure (Fig. 1a) clearly shows that the distribution of SiC in the aluminium matrix is not uniform. The whisker-reinforced material that is extruded and cross-rolled into plate (Fig. 1b) has a more uniform microstructure, although the texture of the SiC in the aluminium matrix is evident as well as preferential alignment of the whiskers in the extrusion direction. The texture of the composite was not affected by HIPping.

Reinforcement affects the aluminium matrix in several important ways. The TEM micrograph of whisker-reinforced material (Fig. 2) shows a high density of dislocations and a very fine subgrain size. The dislocations are a result of differential thermal contraction of the matrix and the carbide during cooling from the processing or heat-treatment temperature [7]. The small subgrain size is a result of low-angle boundaries, usually only several degrees of tilt between each grain, emanating from the corners of the hexagonal cross-section SiC whiskers. The TEM observations are reported elsewhere in more detail, see [9].

Although the extruded and extruded plus cross-rolled materials are more uniform with regards to SiC distribution than the as-pressed material, we did note a preferred alignment of the SiC reinforcement that is a particularly important material feature when discussing the wetting results presented below. As illustrated in the TEM micrograph in Fig. 3, channels are revealed

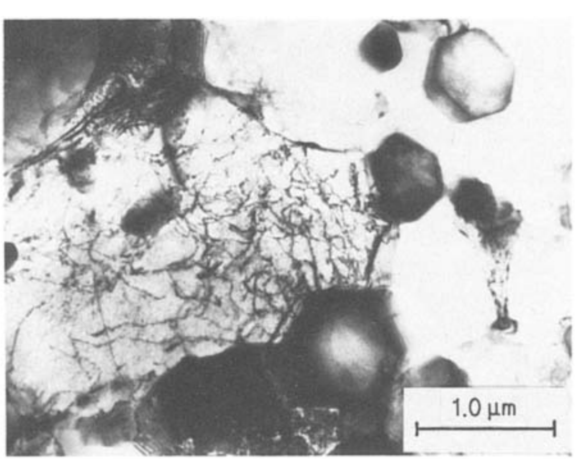


Figure 2 Thin area of a plate of SiC_w/6061 Al in the as-received condition. The foil was cut normal to the extrusion direction, illustrating the low-angle boundaries emanating from the corners of the hexagonally shaped SiC whiskers. The grain size is of the order of several micrometres in this direction.

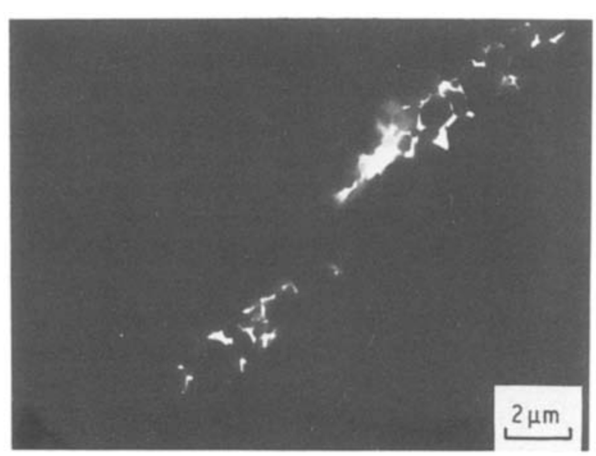


Figure 3 Two carbide-lined channels aligned in the extrusion direction with the minor axis in the short transverse direction of the plate.

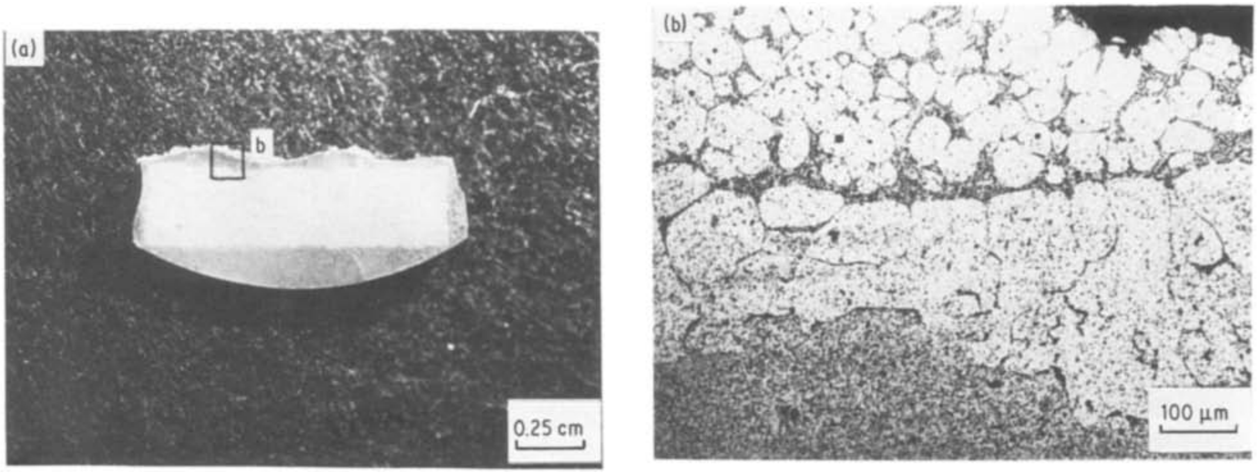
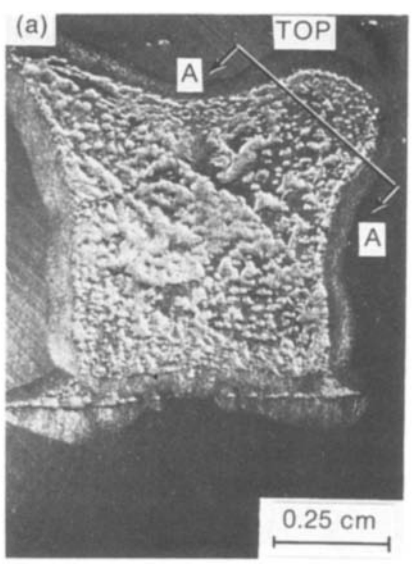


Figure 4 Cross-section of wetting test: (a) Al–Si eutectic alloy on 6061 Al plate after heating to 585° C at 1 mPa, and (b) micrograph of the interface between the Al–Si alloy and the 6061 Al showing alloying and grain-boundary penetration.

in regions well away from the thin areas of the ion-milled sample. The channel cross-section is lenticular in shape, with the minor axis in the through-thickness direction of the plate. The channels may be caused either by voids in the as-pressed composite that become elongated during extrusion or preferential milling of aluminium-rich areas while preparing the sample for TEM. In either case, they represent a texture in the material that may be partially responsible for the observed eutectic alloy interactions.

3.2. Wetting tests
3.2.1. Al–Si eutectic alloy
In initial wetting tests conducted on wrought 6061-T6 Al coupons using the Al–Si eutectic alloy, the alloy flowed over the surface of the coupon at 590° C, eventually covering the entire sample. The cross-sections seen in Fig. 4 illustrate the excellent wettability of the alloy on 6061 Al. The Al–Si eutectic did alloy over a narrow region at the interface between it and the 6061 Al. Limited alloying is a necessary step in the formation of a braze joint. The depth of penetration of the Al–Si was not uniform along the interface and entered somewhat further into the 6061 Al near the grain boundaries (Fig. 4).

Similar wetting tests on extruded SiC_w/Al coupons indicated that the Al–Si penetrated deeply into the composite with little flow over the surface (Figs 5a, b). The penetration extended nearly halfway through the sample (1.5 mm) and was accompanied by a marked



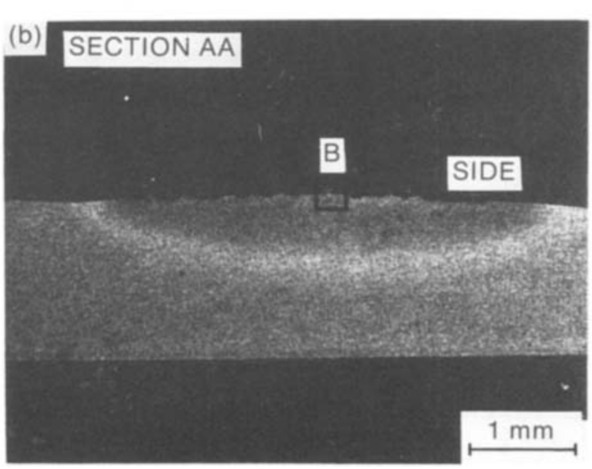
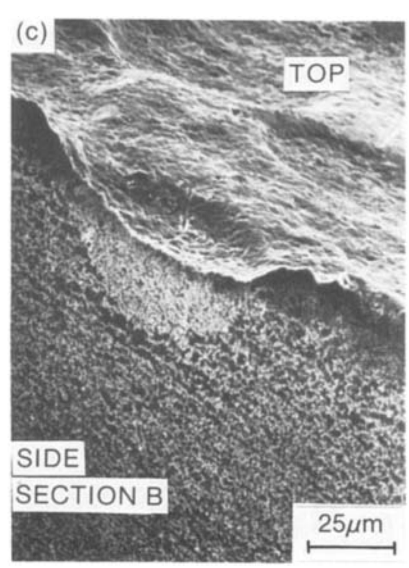


Figure 5 Wetting test with Al–Si eutectic alloy at 590° C: (a) top section of the SiC/Al plate after the wetting test, (b) cross-section of segment A-A revealing the penetration of the Al–Si into the SiC/Al, and (c) high concentration of SiC whiskers collected at the eutectic alloy/composite interface.



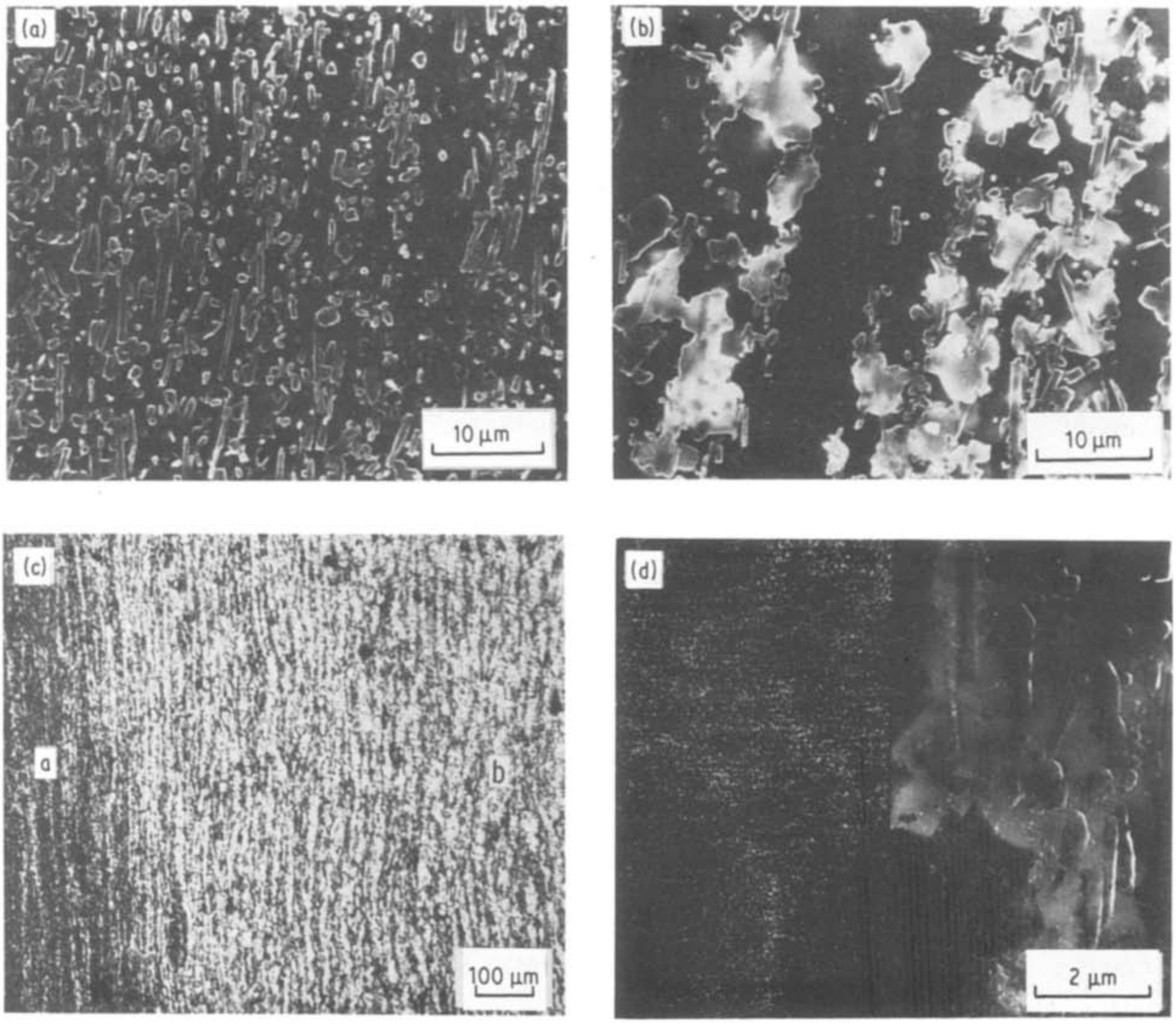


Figure 6 Detailed micrographs of an Al–Ge wetting test at 450° C: (a) electron micrograph of bulk material showing uniformly distributed SiC whiskers, and (b) the reacted composite region; (c) optical micrograph of the transition region substrate at left, the Al–Ge alloy on right, and (d) germanium X-ray map and corresponding electron micrograph.

change in the composite microstructure. After penetration, the SiC whiskers separated into lamellae, with regions rich in carbides separated by areas that were virtually carbide-free (Fig. 5c). In addition, the top-most part of the composite was enriched in SiC, indicating that during the re-solidification of the aluminium matrix alloy, the carbide is at least partially rejected from the melt in the temperature regime of the wetting tests.

3.2.2. Al–Ge eutectic alloy

The Al–Ge eutectic alloy exhibited behaviour similar to the Al–Si alloy even though the tests were run at a much lower temperature (450° C). The Al–Ge system provides more flexibility in running tests as a function of temperature because the eutectic temperature is well below that of the 6061 Al alloy. In addition, germanium in the area exhibiting penetration using the SiC whiskers, so that by measuring the location of germaniun in the area exhibiting penetration using energy dispersive X-ray analysis (EDS), we are able to determine where the eutectic alloy resides in the microstructure. In the case of the Al–Si alloy, this determination becomes difficult because the silicon in the SiC and in the 6061 Al matrix cannot easily be differentiated from the silicon in the Al–Si alloy.

Figs 6a to c show the interface between the composite that interacted with the Al–Ge alloy and the composite away from the affected area. Like the Al–Si alloy, the spacing between the SiC-rich regions and areas nearly devoid of whiskers is accentuated in the area where the Al–Ge has penetrated into the composite. Results of EDS analysis on this affected area indicated that germanium-rich material segregated to the SiC_w-rich region (Fig. 6d).

Several further tests were performed using the Al–Ge system. In one experiment, composite material was subjected to a wetting test using the Al–Si eutectic system and then re-tested using the Al–Ge system. The results of this test (Fig. 7) demonstrated that the first experiment with Al–Si resulted in substantial interaction between the alloy and the composite. However, the Al–Ge did not penetrate into areas of the composites that were already affected by the Al–Si alloy. Instead the Al–Ge alloy flowed along the composite surface until it reached an area that had not been affected by the Al–Si and then penetrated into the composite.

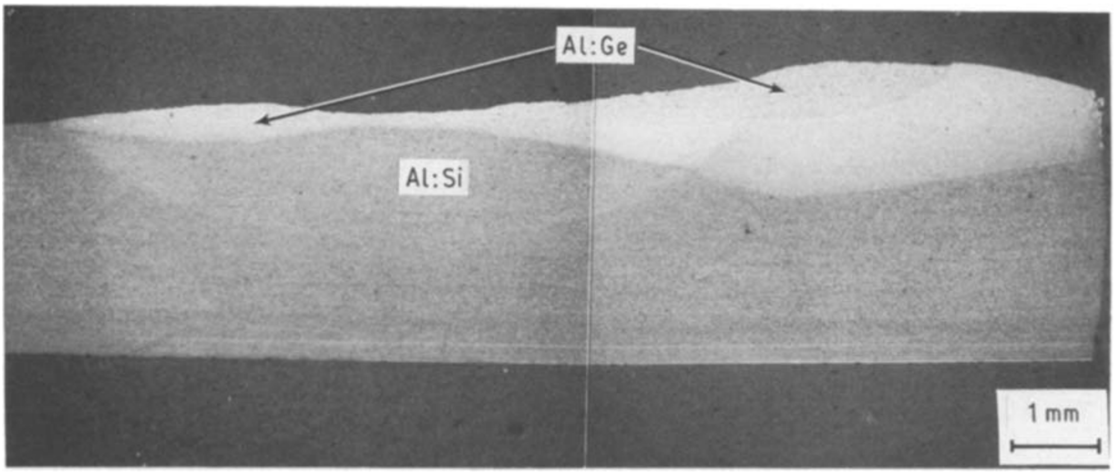


Figure 7 Effect of temperature on the penetration of a eutectic mixture of aluminium and zinc on SiC/6061 Al. Little penetration is evident, even at temperatures where Al–Ge rapidly attacks the composite.

3.2.3. Zn–Al eutectic alloy
Unlike the other alloys, the zinc-based alloy did not penetrate the composite but instead flowed over its surface with only a limited interaction zone. Tests as a function of temperature (Fig. 8) indicated little change in the degree of penetration even at temperatures where the Al–Ge alloy exhibited marked penetration (462° C).

Wetting of the composite improved with increasing temperature as evidenced by a decreasing contact angle and greater surface coverage. A loss of eutectic alloy was noted at the higher temperatures, undoubtedly caused by zinc vaporization in the vacuum furnace [10].

Examination of the interaction between the Zn–Al alloy and the composite (Figs 9a to c) revealed a limited alloying region, similar to that observed between the Al–Si alloy and 6061 Al (Fig. 4). The penetration zone did not exhibit lamellae; instead it solidified into a cellular structure. The zinc in the interface zone was located using back-scattered electron microscopy which revealed that the SiC (dark phase) was rejected by both the zinc-rich (bright-area) and aluminium-rich (grey-area) phases during solidification, as evidenced by whiskers in the areas that were the last to freeze (Fig. 9d).

3.3. Composite microstructure effects
The penetration of Al–Si and Al–Ge into the composite and the subsequent formation of a lamellar structure in the extrusion direction suggests that the materials processing history plays a strong role in the eutectic alloy/composite interaction. To study the effect of processing history on eutectic alloy penetration, experiments were performed on materials in the hot-pressed condition, in the extruded and cross-rolled condition and T6 heat treatment, and in the extruded, cross-rolled and HIPped condition.

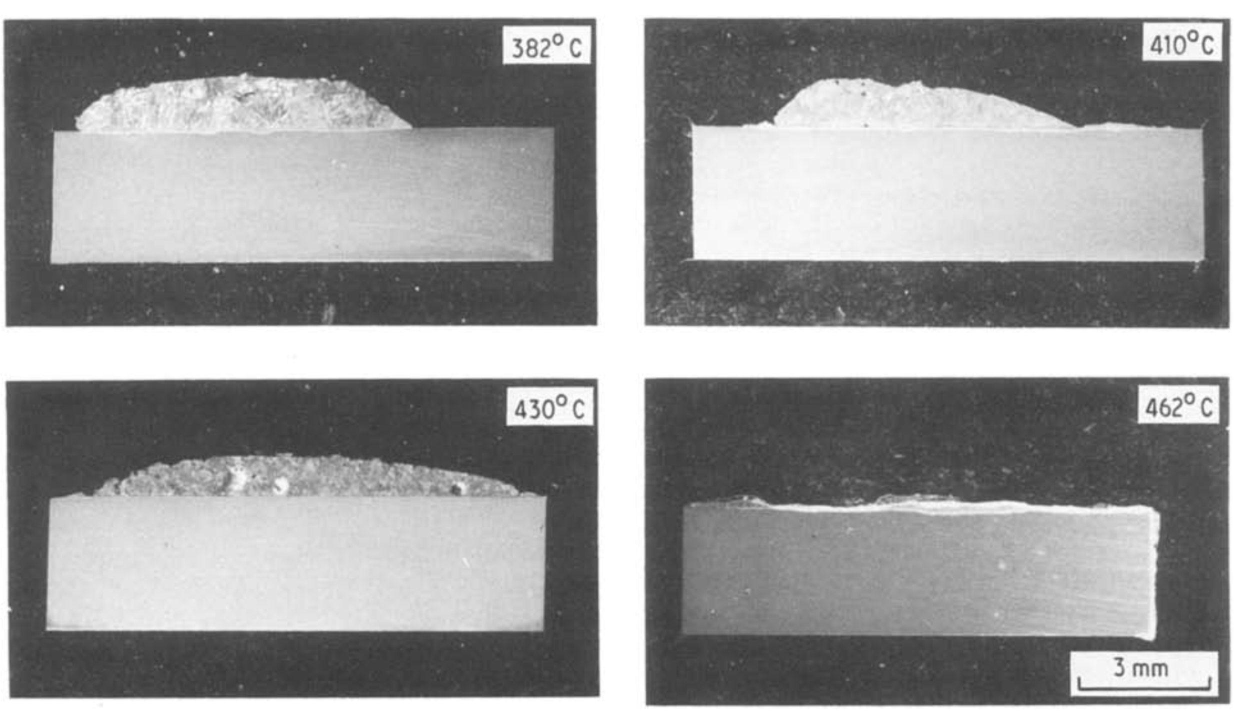


Figure 8 The wetting of SiC_w/Al by Zn–Al for temperatures ranging from 382 to 462° C. Wetting improves as a function of temperature.

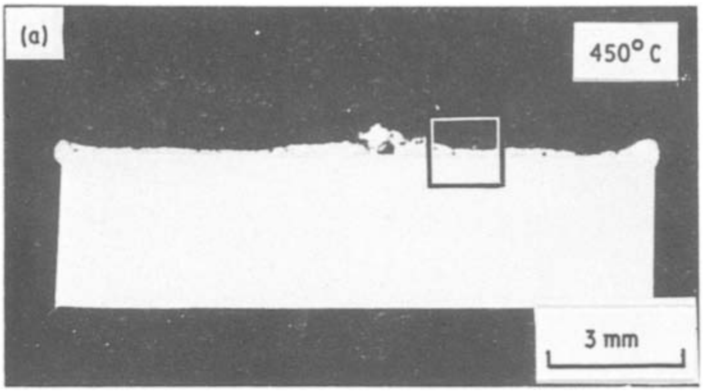
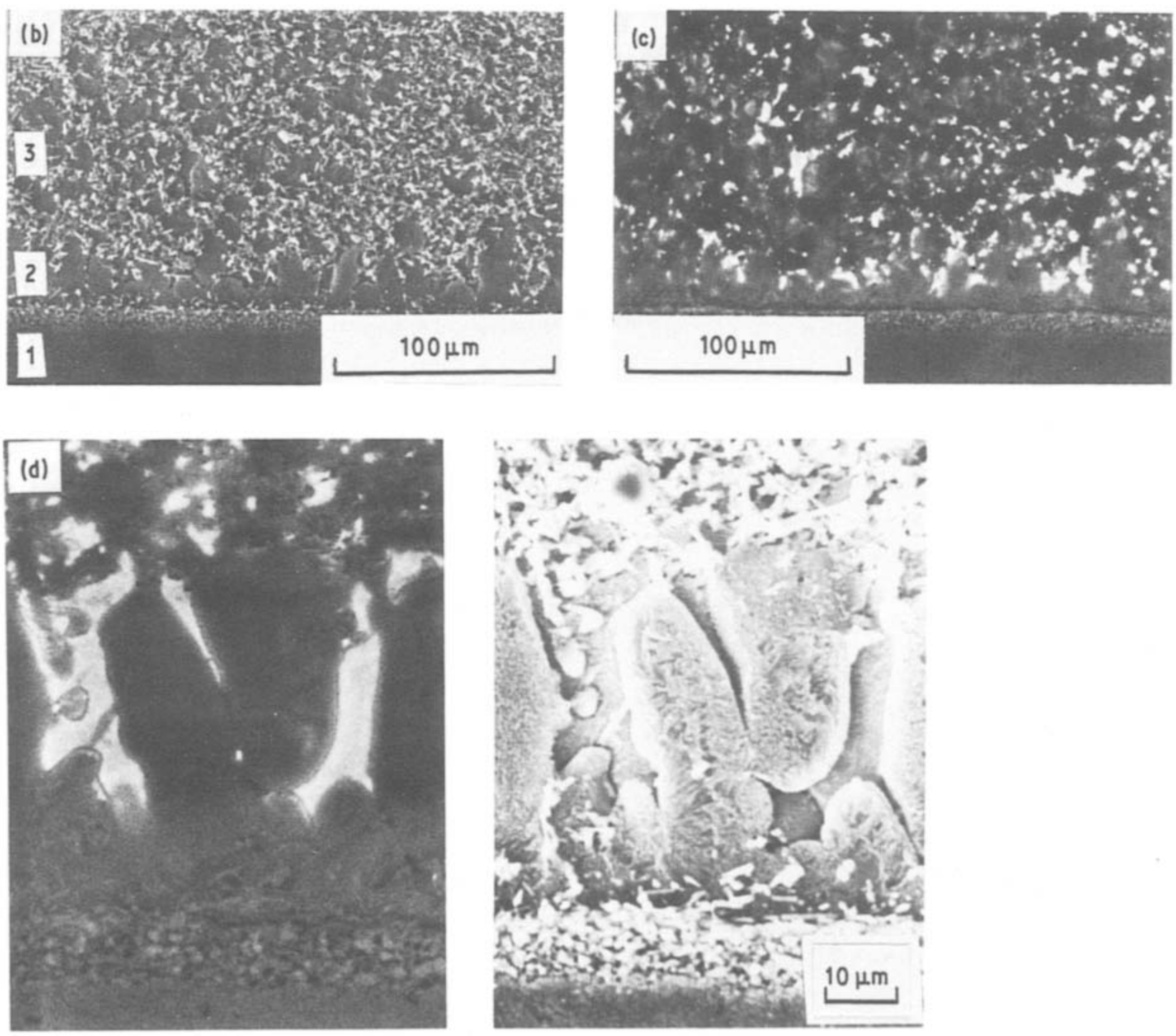


Figure 9 Wetting test results on extruded SiC_w/Al plate at 450° C for 30 sec using eutectic Zn–Al: (a) cross-section of wetting test sample; (b) blow-up of square area in (a) at interface; (c) backscatter electron micrograph of (b); and (d) cellular structure at interface (backscatter electron micrograph on the left, and secondary electron micrograph on the right).



In the case of the hot-pressed material, the composite exhibits prior boundaries of the aluminium powder and the interior of the aluminium particles are devoid of SiC, as illustrated earlier (Fig. 1a). The distance between the SiC particles at the aluminium–powder boundary is similar to that of the extruded material. These results of the wetting tests demonstrated that the Al–Si and Al–Ge alloys did not penetrate this material (Fig. 10).

In the case of the HIPped material, the composite exhibited little change in microstructure from the extruded material. The material was HIPped in order to close any possible internal porosity or channels that might have been formed during the working process. Wetting tests demonstrated that the Al–Si eutectic penetrated the composite in a similar fashion as that previously discussed for the extruded composite that had not been HIPped. The microstructural effects are summarized in Table II.

TABLE II Processing effects on SiC_w/Al resistance to attack

Processing condition	Eutectic alloy		
	Al–Si	Al–Ge	Zn–Al
Hot-pressed billet	W	W	W
Extruded and cross-rolled	A	A	W
Extruded and cross-rolled plus HIPped	A	A	W

A: Molten eutectic alloy deeply penetrates into the composite.
W: Composite wet by the molten eutectic alloy with limited alloying between the two.

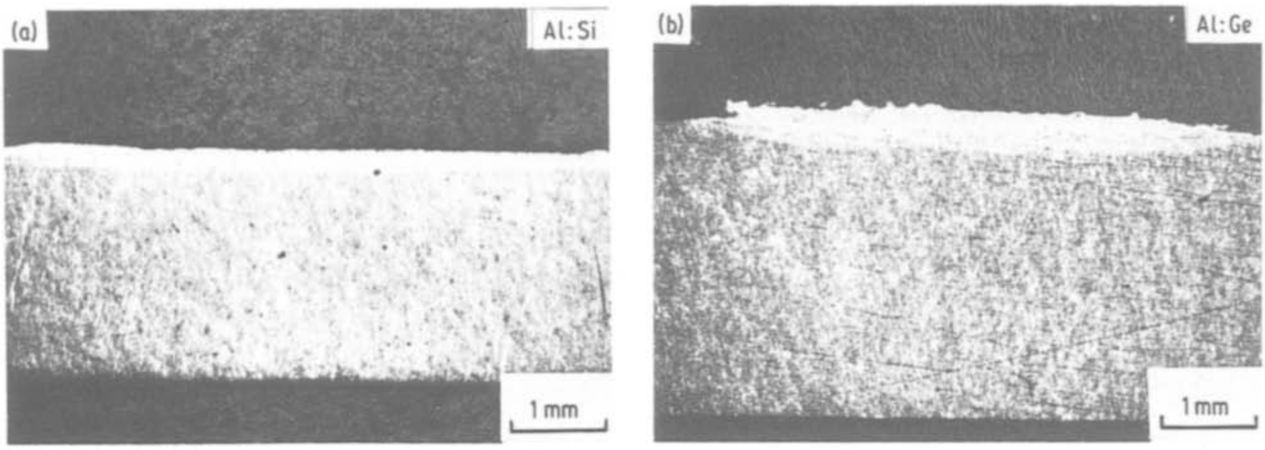


Figure 10 Result of interaction of hot-pressed SiC_w/Al billet with (a) the eutectic Al–Si alloy, and (b) the eutectic Al–Ge alloy. Neither alloy penetrated the hot-pressed billet.

4. Discussion

The results from the previous section indicate that several mechanisms may contribute to the penetration of certain eutectic alloys into the composite. Both the Al–Si and the Al–Ge alloys were found to rapidly penetrate the extruded and cross-rolled whisker-reinforced composite, whereas the Zn–Al alloy acted more conventionally, in that it reacted slowly with the composite to form a thin metallurgical interface. In particular, wetting tests show that the Al–Ge alloy penetrates deeply into the composite at the temperature at which the Zn–Al alloy only wets the surface.

The penetration of the eutectic alloys appears to be related to their ability to wet the SiC whiskers. The mode of penetration into the composite for the Al–Si and Al–Ge alloys differs greatly from that of the Zn–Al alloy. In the case of the former two, the homogeneous SiC whisker structure of the composite broke down into a lamellar structure consisting of SiC-rich regions, separated by an area almost totally whisker-free. The whisker-free regions also contained high concentrations of silicon and germanium from the eutectic alloy. The SiC-rich lamellae ran normal to the surface of the plate and were separated by aluminium-rich regions. The spacing between lamellae was approximately 10 μm. For the Zn–Al alloy, no lamellar structure was found. Penetration by the Zn–Al into the composite was slow and the last areas to solidify contained the greatest density of whiskers. The mode of attack by the different eutectic alloys and the resulting microstructure suggests that wetting of the SiC by the eutectic alloy is an important factor in the penetration of the alloy into the composite.

Another variable that appears to play a significant role in the reaction of the eutectic alloys is the processing history of the composite. Although both Al–Si and Al–Ge reacted strongly with the extruded and cross-rolled material, it did not attack similar material that had not been worked. Penetration by these two eutectic alloys was very shallow in the as-pressed billets, suggesting that the working operation may have formed some preferential path in the composite. The banded structure that resulted during ion milling of the thin foils is a result of differential sputtering which could be due to changes in the concentration of aluminium in the composite. Working the material does not entirely homogenize the whisker distribution in the composite, but leaves behind areas relatively rich in SiC alternating with areas rich in aluminium.

In summary, in order for rapid penetration of the composite to occur, two conditions appear necessary. The composite must be heavily worked and the eutectic alloy must wet the SiC whiskers. Therefore, the rapid penetration appears to be caused by the flow of the eutectic alloy down preferential paths in the composite. The resulting lamellae in the extrusion direction and the presence of the banded structure in the thinned foils of the same dimensional spacing as the lamellae suggests a compositon difference in the material arising during the working operation. In addition, preference of silicon and germanium to the SiC-rich lamellae, and the absence of such a preference for the Zn–Al alloy, suggests that the rapid penetration may result from the wettability of SiC by the constituents in the eutectic alloy.

Acknowledgements

The authors would like to thank Brian Quigley at the Naval Surface Weapons Center in Silver Spring, Maryland for the support of this work and Jim Early at the National Bureau of Standards in Gaithersburg, Maryland for use of their HIPping facility. This work was performed under NSWC contract no. N60921-83-C-0154.

References

1. J. S. AHEARN, D. C. COOKE and S. G. FISHMAN, *Metal Construction* **14**(4) (1982) 192.
2. J. S. AHEARN and D. C. COOKE, The Weldability of SiC/Al Metal Matrix Composites, MML-TR-82-15c (1982).
3. J. S. AHEARN, D. K. McNAMARA and D. C. COOKE, Weldment Design for a SiC/Al SPS-10 Antenna, NSWC Quarterly Report on contract N60921-82-C-0067 (1982).
4. G. F. PITTINATO, Inertia Welding of Metal Matrix Composites Lightweight Torpedo Hull Structure (McDonnel Douglas Astronautics Co., Huntington Beach, California, Report No. MDC-H1034-1, NSWC Contact No. N60921-83-C-0176, Silverspring, Maryland, 1984).
5. J. S. AHEARN, D. C. COOKE and E. BARTA, Joining Discontinous SiC/Al Metal Matrix Composites, MML-TR-85-44c (1985).

6. J. S. AHEARN, W. C. MOSHIER and D. C. COOKE, Brazing of SiC/Al Composites, MML-TR-84-53c (1984).
7. W. A. ANDERSON, "The Effect of Metallurgical Structure on the Brazing of Aluminium", Proceedings TMS-AIME Physical Metallurgy of Metal Joining, edited by R. Kossowky and M. E. Glicksman (AIME, Warrendale, Pennsylvania, 1980) pp. 222-244.
8. K. F. DOCKUS, *Welding J.* **57**(10) (1978). 36.
9. J. S. AHEARN and D. C. COOKE, to be published.
10. P. McNAMARA and O. R. SINGLETON, *Welding J.* **58**(1) (1979) 17-2.

Received 10 February
and accepted 10 April 1986