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Trans. Nonferrous Met. Soc. China 22(2012) 596−603

Transactions of Nonferrous Metals Society of China

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Development of Al−12Si−xTi system active ternary filler metals for Al metal matrix composites

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Received 4 March 2011; accepted 26 June 2011

Abstract: To improve the wettability of Al metal matrix composites (Al-MMCs) by common filler metals, Al−12Si−xTi (x=0.1, 0.5, 1, 3.0; mass fraction, %) system active ternary filler metals were prepared. It was demonstrated that although the added Ti existed within Ti(Al$_{1-x}$Si$_x$)$_3$ ($0\le x\le 0.15$) phase, the shear strength and shear fracture surface of the developed Al−12Si−xTi brazes were quite similar to those of traditional Al−12Si braze due to the presence of similar microstructure of Al−Si eutectic microstructure with large volume fraction. So, small Ti addition (~1%) did not make the active brazes brittle and hard compared with the conventional Al−12Si braze. The measured melting range of each Al−12Si−xTi foil was very similar, i.e., 580−590 °C, because the composition was close to that of eutectic. For wettability improvement, with increasing Ti content, the interfacial gap between the Al_2O_3 reinforcement and filler metal (R/M) could be eliminated, and the amount of the remainder of the active fillers on the composite substrate decreased after sessile drop test at 610 °C for 30 min. So, the wettability improvement became easy to observe repeatedly with increasing Ti content. Additionally, the amount and size of Ti(AlSi)$_3$ phase were sensitive to the Ti content (before brazing) and Si content (after brazing).

Key words: Al metal matrix composites; brazing; transient liquid phase bonding; wettability; filler metal

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1 Introduction

Compared with fusion welding processes, both transient liquid phase (TLP) diffusion bonding and brazing do not involve the melting of the base metals. So, the problems of reinforcement-metal (R/M) reactions, reinforcement destruction, etc, can be reduced [1]. Currently, TLP bonding and brazing for Al metal matrix composites (Al-MMCs) are generally accepted as commercial practicable methods instead of fusion welding processes. The principal factor governing the interfacial microstructure and properties of TLP bonded or brazed joints is the alloying system selection and corresponding composition design of interlayer or brazing filler metal, which determines the removal of oxide film, particle segregation and wettability of Al-MMCs by filler metals. In previous reports, Al−12Si brazing filler metal [2] and Zn−Al eutectic solder [3−5] have been used in the studies of brazing of Al-MMCs, while several pure materials such as Cu foil [6−9], Ag foil [10] and Si powders [11] were used as interlayer in TLP bonding of Al-MMCs or monolithic Al to effectively remove oxide film on Al matrix surface by undermining mechanism [4, 12].

As improved interlayers, Al−Cu alloy interlayer [13] and Cu/Al/Cu sandwich interlayer [14], Cu/Ni/Cu [15] sandwich interlayer, and Al−Si−Ti−SiC mixed powders [16] were used to reduce particle segregation, to reduce bonding temperature, and to get a bond seam with reinforcement, respectively. On the other hand, as improved process for TLP bonding, such as temperature gradient TLP bonding (TG-TLP) [17], TLP bonding followed by isostatic pressing [18], mechanical action assisted TLP bonding (introducing ultrasonic vibration [19], electric [20] or impact [21]), semi-solid state TLP bonding [22,23], and quasi-melting [24,25] were attempted to make the profile of bond line flexural, to improve the interface densification with a minimal deformation, or to effectively remove tenacious oxide

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Foundation item: Project (50875199) supported by the National Natural Science Foundation of China; Project supported by State Key Laboratory of Advanced Welding and Joining, China

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DOI: 10.1016/S1003-6326(11)61219-3

film on Al matrix surface.

However, the popularly used Al−12Si filler metal [2], Al−Si−Mg filler metal [26] and Cu interlayer [27] showed poor wettability at R/M micro-interfaces, especially for Al_2O_3 reinforcing particulates, resulting in interfacial gap [2] and interfacial debonding fracture mode at R/M micro-interfaces [27, 28]. Recently, a novel process of active-TLP bonding (A-TLP) was proposed by ZHANG et al [27] to improve the wettability at R/M micro-interfaces, in which the interlayer must contain both melting point depressant (MPD) and active element for ceramic reinforcement (e.g., Ti). Despite numerous efforts made to overcome a specific problem, a generally accepted alloying system selection route and corresponding composition design route for the interlayer or brazing filler metal have not been established.

It should be pointed out that a binary interlayer consisting of MPD and active filler metal (e.g., Ti) can not reduce particle segregation and simultaneously can not improve wettability at R/M interfaces. Therefore, in the present work, Al−12Si−xTi (x=0.1, 0.5, 1.0, 3.0; mass fraction, %) system active ternary filler metals were prepared to reduce particle segregation and simultaneously to improve the wettability of particulate ceramic reinforcement by traditional Al−12Si filler. Their composition design route, melting range, microstructures and properties (including microhardness and shear property), and wettability were described and compared to optimize alloying system selection and corresponding composition design.

2 Experimental

As mentioned above, according to the composition design route of interlayer for A-TLP bonding of Al-MMCs [26, 27], the active ternary filler metal of Al−Si−Ti system was developed, in which Al was added to reduce particle segregation, Si was selected as MPD to remove the oxide film on Al matrix of Al-MMC by undermining, and Ti was added to improve the wettability for Al-MMC through introducing the reactions at filler metal/matrix (M/M) and/or at R/M interface.

To improve the reactive wettability between the active filler metal and Al_2O_3 ceramic reinforcement at TLP bonding temperature (generally about 600 °C), the added Ti should be in molten state, but not in solid state, because the added Ti atoms in liquid state will have high activity and distribute uniformly. However, the high melting point of Ti (1668 °C) and the absence of eutectic reaction in Al−Ti binary system imply that the added Ti may be refractory as a “melting point raiser” for Al matrix. Therefore, the Ti content should be selected carefully to make the added Ti atoms molten and uniform during the A-TLP bonding.

Based on the liquidus projection of Al−Si−Ti ternary phase diagram at Al-rich corner (see Fig. 1) [29,30], in which a Al−Si−Ti ternary eutectic reaction exists for the composition of Al−12Si−1Ti at 573 °C (described as: $L\leftrightarrow(\mathrm{Al})+(\mathrm{Si})+\tau_1$), a series of Al−12Si−$x$Ti ($x$=0.1, 0.5, 1.0, 3.0) system active ternary filler metals with a composition very close to the eutectic composition of Al−12Si−1Ti were selected to completely (or partially) make the added Ti atoms molten and uniform in molten interlayer during A-TLP bonding. So, the reactive wetting both at M/M and R/M micro-interfaces may be expected. The used braze ingot was obtained by melting three kinds of commercially available blocks of pure Al, Al−20Si and Al−5Ti master alloys in flowing argon with high frequency induction heating. The resultant melt was kept at 830 °C for 30 min, and then cooled in situ to room temperature. And their foils were prepared by re-melting the in-situ solidified ingot and then rapid cooling.

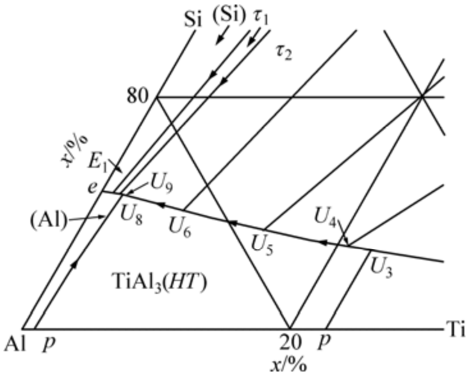

Fig. 1 Liquidus projection of Al−Si−Ti ternary phase diagram at Al-rich corner showing the presence of ternary eutectic reaction [29, 30] (*p*: Peritectic reaction; *e*: Eutectic reaction; *u*: Transition reaction)

As described in detail in a previous studies [10], the used Al-MMC produced by pressure casting method had pure aluminum matrix (to simplify element analysis) and short fiber reinforcement consisting of Al_2O_3 (85%, mass fraction) and SiO_2 (15%). For convenience, the used Al MMC was marked by Al_2O_{3sf}/Al in this paper. The average diameter and average length of the short fiber reinforcement were 3.6 μm and 140 μm, respectively, and the volume fraction of the short fiber reinforcement in the Al_2O_{3sf}/Al composite was 30% [10]. The as-received Al_2O_{3sf}/Al composite block was cut into pieces of 15 mm×15 mm×2 mm. Before each test, the piece surface was polished with 600 emery paper, and then ultrasonically degreased in acetone.

The microstructures and properties of the ingot filler metals (including shear strength and microhardness for different phases) were examined. Especially, the existing form and morphology of Ti in filler metals and the effect of Ti addition on the microstructure and property of filler metals were examined by back-scattered electron (BSE) images and by energy dispersive X-ray (EDX) analysis. The melting range of each Al−12Si−xTi (x=0, 0.1, 0.5, 1.0, 3.0) foil was measured with differential scanning calorimetry (DSC). According to the measured melting range, the wettability of acceptable foils on Al_2O_{3sf}/Al composite was tested by sessile drop method.

3 Results and discussion

3.1 Microstructure of Al−Si−Ti system filler metals and existing form of active element Ti

Figure 2 shows the BSE images of the cross-section of in situ solidified ingots of Al−12Si, Al−12Si−0.1Ti, Al−12Si−0.5Ti and Al−12Si−1Ti, respectively. The microstructures of them are quite similar, mainly consist of α(Al)−Si eutectic. However, with increasing Ti content from 0.1% to 1%, a flake-like white phase is present considerably, which must contain Ti with an atomic number of 22, much higher than that of Al (13) or Si (14). So, the microstructure of Al−12Si−xTi ingot consists of α(Al)−Si eutectic (similar to Fig. 2(a)) and a flake-like phase containing Ti, and the added Ti should be almost within the precipitated phase. This result is consistent with the reports by ZEREN and KARAKULAK [31, 32].

To identify the kind of the white precipitated phase containing Ti and to further investigate the existing form of active element Ti, the composition of the white precipitated phase containing Ti was tested. EDX spot analysis (see Figs. 2(c) and (d)) results show that the light flake-like white phase has a composition of 30% (mole fraction) Ti, 58% Al and 12% Si, which could be identified as a ternary compound of Ti_3Al_5Si. So, the flake-like white precipitated phase belongs to a Al-rich phase of $Ti(Al_{1-x}Si_x)_3$ ($0\le x\le 0.15$), commonly written as $Ti(AlSi)_3$ or τ_3 [32], in which up to 15% Al can be replaced by silicon in $TiAl_3$ lattice structure, resulting in various chemical compositions and a range of lattice parameters. It can also be seen from Fig. 2 that the size of the present $Ti(AlSi)_3$ phase is larger than that of Si phase, and both the number and size of the precipitated $Ti(AlSi)_3$ phase increase with increasing Ti content. For Al−12Si−1Ti, the size of $Ti(AlSi)_3$ phase is 100−200 μm in length and 10−20 μm in width within the cross-section (see Fig. 2(d)). So, large size of the $Ti(AlSi)_3$ phase is not beneficial to the ductility of the braze and leads to nonuniform distribution of Ti (especially for small content of about 1%) at each micro-area. So, the filler metal should be used in foil obtained by rapid cooling process to reduce precipitation and to improve the distribution of Ti in solid filler metal.

To further investigate the mechanical property of the $Ti(Al_{1-x}Si_x)_3$ phase ($0\le x\le 0.15$), the Vickers microhardness testing was carried out on distinct phases including Al, Si, and $Ti(Al_{1-x}Si_x)_3$ in the Al−12Si−1Ti ingot at a load of 24.5 mN for a dwell time of 10 s (HV_{25g}). Figure 3 shows the indentation on distinct phases in Al−12Si−1Ti ingot and corresponding Vickers microhardness, respectively. As expected, the $Ti(Al_{1-x}Si_x)_3$ phase is much harder (HV_{25g} 363) than Al

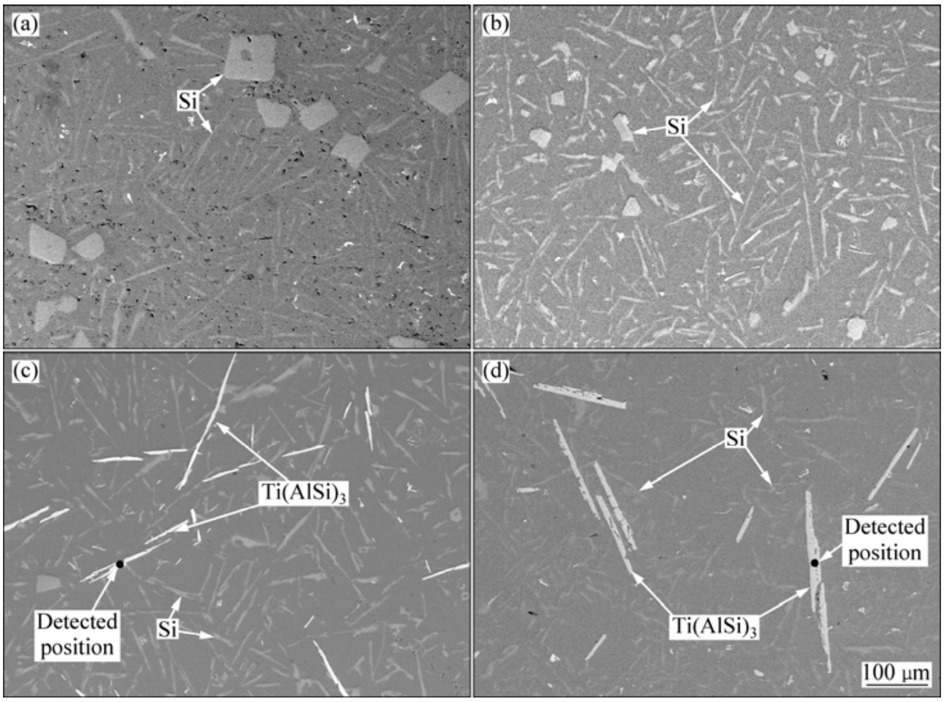

Fig. 2 BSE images of in situ solidified filler metal ingots: (a) Al−12Si; (b) Al−12Si−0.1Ti; (c) Al−12Si−0.5Ti; (d) Al−12Si−1Ti

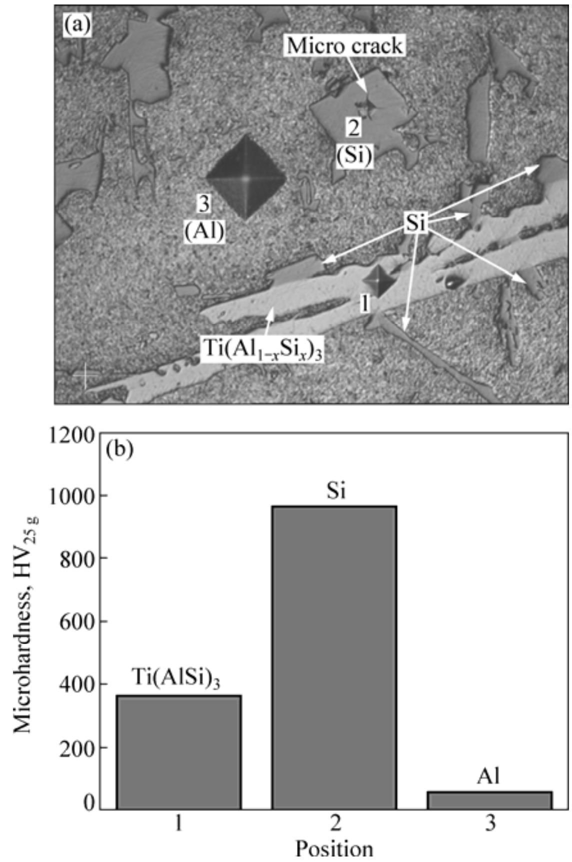

Fig. 3 Micro-indentation test on distinct phases in Al−12Si−1Ti ingot (a) and their microhardness (24.5 mN, 10 s) (b)

substrate (HV_{25g} 56). On the other hand, micro crack is observed only on Si phase, showing that the hard $Ti(Al_{1-x}Si_x)_3$ phase is not brittle as Si phase. In fact, the $Ti(AlSi)_3$ phase may be a promising reinforcement for Al−Si−*x*Ti cast alloy, which is used as a wear-resistant material [32]. Moreover, it is interesting that some Si is in contact with the $Ti(Al_{1-x}Si_x)_3$ phase, suggesting that some Si particles possibly nucleate at $Ti(AlSi)_3$.

3.2 Shear strength and fracture surface of Al−12Si−*x*Ti ingots

Figure 4 shows the shear strength values of machined ingot samples of Al−12Si and Al−12Si−*x*Ti (*x*=0.1, 0.5, 1.0) with the same size (cross section: 5 mm×5 mm; length: 15 mm). The shear strength values of Al−12Si−*x*Ti (*x*=0.1, 0.5, 1) samples are quite similar to each other, and are also quite similar to that of the Al−12Si sample. The measured shear strength, with small scatter, is about 100 MPa. Figure 5 shows the

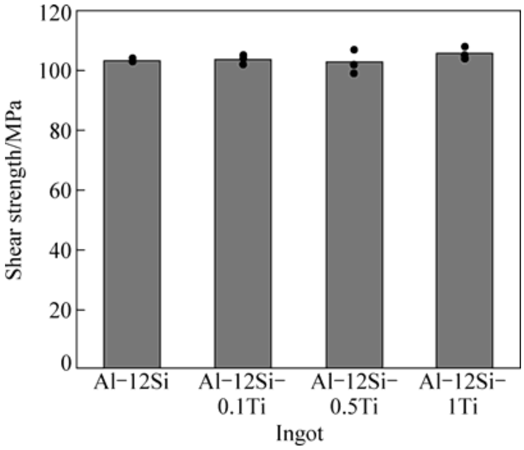

Fig. 4 Shear strength of Al−12Si and Al−12Si−*x*Ti (*x*=0.1, 0.5, 1.0) filler metals ingots

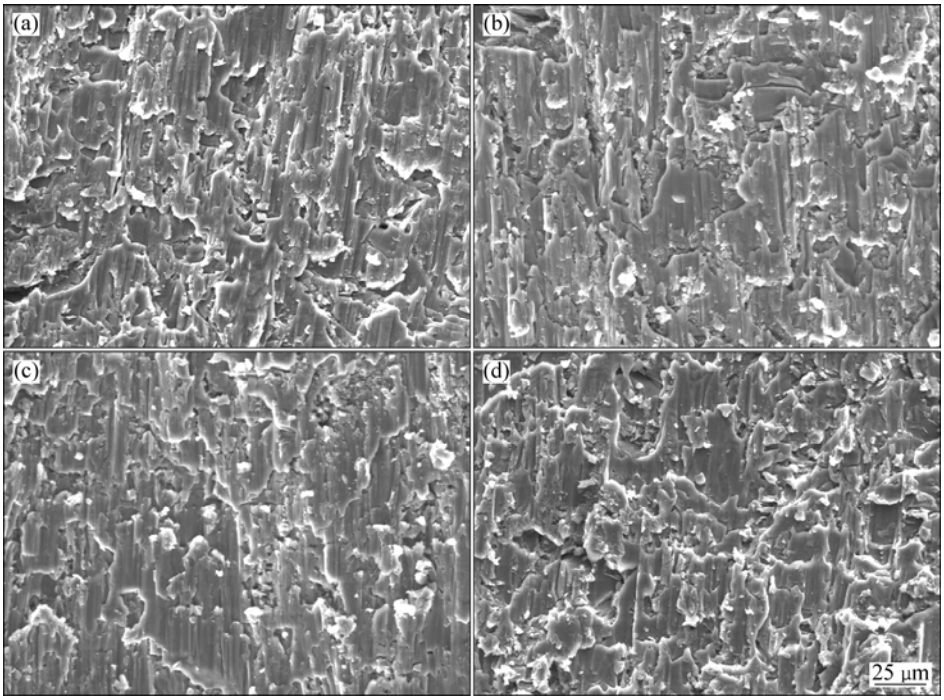

Fig. 5 Shear fracture surfaces of Al−12Si (a), Al−12Si−0.1Ti (b), Al−12Si−0.5Ti (c) and Al−12Si−1Ti (d) ingots

corresponding fracture surfaces of Al−12Si and Al−12Si−*x*Ti (*x*=0.1, 0.5, 1.0) samples after shear test. Like Al−12Si sample, significant plastic deformation marks can be observed on the shear fracture surface of each developed Al−12Si−*x*Ti samples.

Based on the fact that both shear strength and shear fracture surface of each Al−12Si−*x*Ti (*x*=0.1, 0.5, 1) are quite similar to those of the Al−12Si ingot, it can be deduced that small Ti addition (~1%) does not make the novel brazes brittle and hard compared with the conventional Al−12Si braze. The similar shear strength and similar shear fracture surface should be a result of similar microstructure. Additionally, it should be noted that although small addition of Ti does not affect shear strength and shear fracture surface of the braze ingots significantly, and the amount of $Ti(AlSi)_3$ phase is sensitive to the Ti content.

3.3 Melting temperature range of braze foils

To make the distribution of small added Ti uniform via reducing the gravity segregation and to make the pre-placement of the developed fillers convenient, the Al−12Si−*x*Ti (*x*=0, 0.1, 0.5, 1.0, 3.0) master alloys were remelted and then rapidly cooled to fabricate braze foils. All of them showed good ability to form brazing foil during rapid cooling process.

Figure 6 shows the DSC curves of conventional Al−12Si foil and the developed Al−12Si−*x*Ti foils (*x*=0.1,

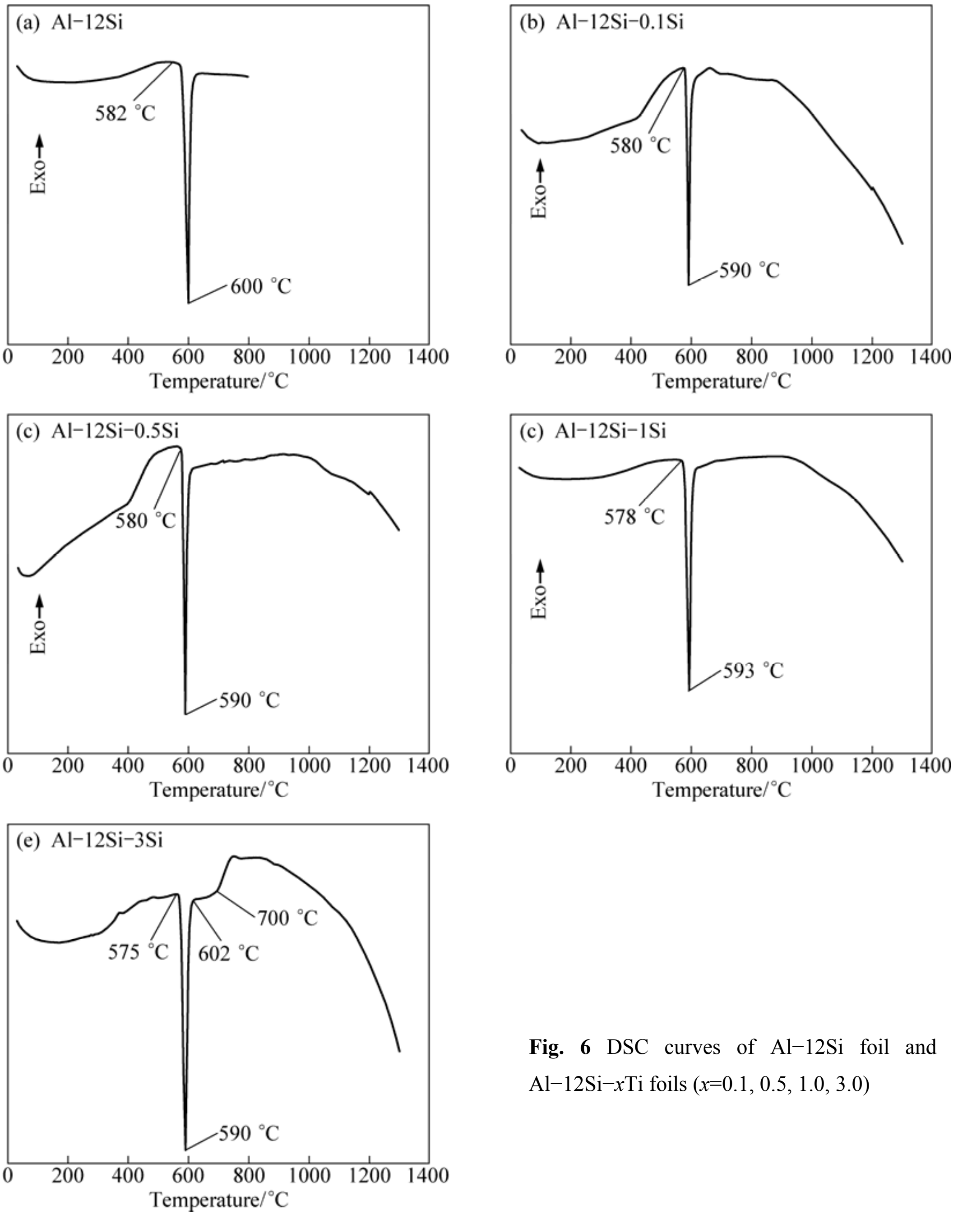

Fig. 6 DSC curves of Al−12Si foil and Al−12Si−*x*Ti foils (*x*=0.1, 0.5, 1.0, 3.0)

0.5, 1.0, 3.0). Like Al−12Si foil, the developed Al−12Si−*x*Ti (x=0.1, 0.5, 1, 3) braze foils also exhibit only one endothermic peak, showing that they should be near ternary eutectic filler metal. It can be seen that the solidus and liquidus temperatures of the developed Al−12Si−*x*Ti filler metal are about 580 °C and 590 °C, respectively. Compared with the melting point of Al−12Si foil, the solidus and liquidus temperatures do not increase when Ti is up to 1%. When the Ti content increases even to 3%, no significant increase in melting range is observed, showing that the effect of Ti content on melting point for Al−Si−Ti ternary is not as sensitive as that for Al−Ti binary system, in which the melting point of Al−Ti binary alloy sharply increases with small Ti addition. Therefore, when bonding temperature is above 600 °C, the added Ti should be in liquid state, and thus be present uniformly at whole joint interface and further can easily react with oxide film on Al matrix surface and/or ceramic reinforcement at whole joint interface.

3.4 Wettability test

Figure 7 shows the interfacial microstructure (BSE images) of the cross section of the wettability testing samples for Al−12Si foil and Al−12Si−*x*Ti foils (x=0.1, 0.5, 1.0) after sessile drop test at 610 °C for 30 min. No particulate segregation occurs for Al−12Si. For conventional Al−12Si foil, a continuous layer slightly thinner than initial thickness of the foil (100 μm) remains on the surface of the composite substrate due to limited penetration of the molten braze into the composite; moreover, although the interfacial gap at M/M micro-interfaces disappears, the interfacial gaps at R/M micro-interfaces could not be eliminated, especially at the areas with numerous short alumina fibers. Generally speaking, the Al_2O_{3sf}/Al composite will show poorer wettability because the pure Al matrix does not contain active Mg element and the Al_2O_3 reinforcement is more stable than SiC in contact with molten Al.

In contrast, in most cases, the interfacial gaps at R/M micro-interfaces could be eliminated finally when small Ti is added, even less than 1%, showing that the developed Al−12Si−*x*Ti active braze could react with Al_2O_3 reinforcement to some extent compared with traditional Al−12Si braze; moreover, the amount of the remainder of the active braze foil decreases with increasing Ti content, showing that with increasing Ti content, the penetration of the Si atoms in the molten brazes into the composite substrate could occur earlier. However, since the Si atoms in the molten braze foil penetrate into the composite substrate earlier with increasing Ti content, resulting in significant decrease in Si content in the remainder of braze foil, the $Ti(AlSi)_3$ phase is present significantly on the surface of remainder of braze foil with increasing Ti content.

For example, Al−12Si−1Ti braze foil completely penetrates into the Al_2O_{3sf}/Al composite, except several light flake-like phases of $Ti(AlSi)_3$ having a composition of 28% Ti, 57% Al and 15% Si. The significant penetration and resultant increase in $Ti(AlSi)_3$ area fraction show that the developed Al−12Si−1Ti filler metal foil could react with the Al_2O_{3sf}/Al composite earlier than conventional Al−12Si braze foil, thus earlier creating some penetration paths without oxide film barrier for Si and then accelerating subsequent undermining. Moreover, bare Al_2O_3 reinforcements are

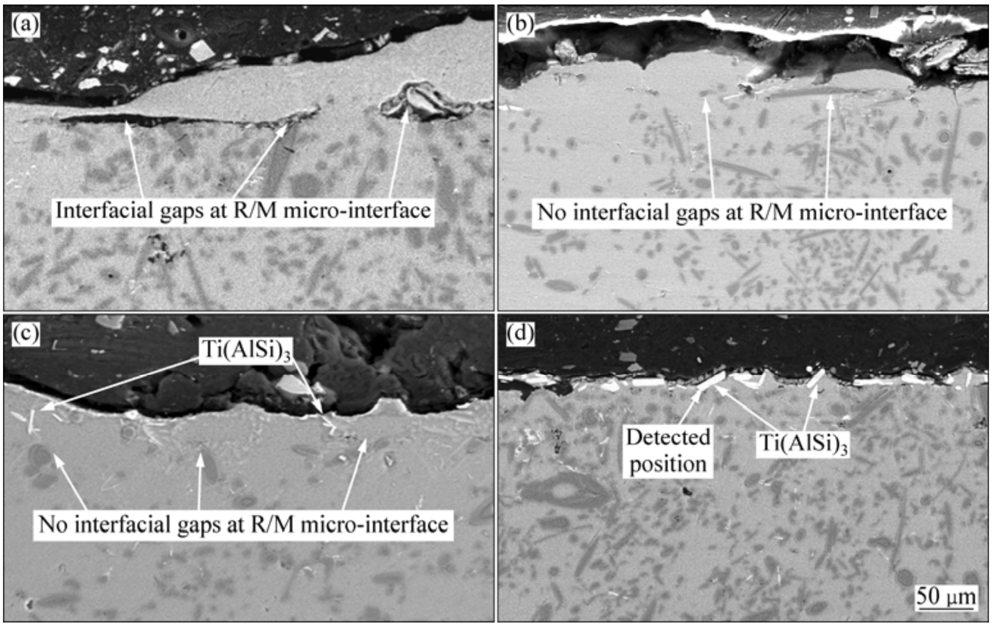

Fig. 7 Interfacial microstructures (BSE images) of cross section of wettability testing samples after sessile drop test at 610 °C for 30 min: (a) Al−12Si; (b) Al−12Si−0.1Ti; (c) Al−12Si−0.5Ti; (d) Al−12Si−1Ti

too hard to observe on the whole surface (with the overall width of 5 mm) after sessile drop test. The results show that the Al_2O_3 reinforcements on initial surface could be covered by the Al−12Si−1Ti filler metal without interfacial gaps. Therefore, the wettability between R/M micro-interface can be improved by Ti addition to traditional Al−12Si filler metal.

It should be noticed that although Ti hardly diffuses into Al matrix of the Al_2O_{3sf}/Al composite and appears only on the upper surface in the form of flake-like $Ti(AlSi)_3$ phase, the maximum size of the $Ti(AlSi)_3$ phase become to be about 25 μm in length and 5 μm in width after wettability test. Especially, the length of the $Ti(AlSi)_3$ phase decreases from 100−200 μm (in initial solid ingot) to 20−30 μm due to the decease in Si content. Moreover, the flake-like $Ti(AlSi)_3$ phases are found to be parallel to the surface of the composite substrate after sessile drop test. If the arranging direction, size and volume fraction of the $Ti(AlSi)_3$ phase with high hardness (HV_{25g} 363) could be controlled properly, the $Ti(AlSi)_3$ phase may be used as a reinforcement to enhance the joint seam.

4 Conclusions

1) The added Ti existed within a flake-like precipitated phase of $Ti(AlSi)_3$, which had a length of 100−200 μm (much longer than eutectic Si phase) and a microhardness of HV_{25g} 363 (much higher than Al phase of HV_{25g} 56). The microstructure of Al−12Si−xTi (x=0.1, 0.5, 1.0) ingot consisted mainly of α(Al)−Si eutectic with $Ti(AlSi)_3$ phase, and both the number and size of the precipitated $Ti(AlSi)_3$ phase increased with increasing Ti content.

2) Compared with the traditional filler metal of Al−12Si, although $Ti(AlSi)_3$ was present, the melting temperature range (580−590 °C), shear strength (~100 MPa) and ductile shear fracture surface of each Al−12Si−xTi (x=0.1, 0.5, 1.0) braze ingots were quite similar to that of the Al−12Si ingot. The results showed that small Ti addition did not increase the melting point or make the novel brazes brittle. These similar characteristics should be a result of similar microstructure.

3) Compared with the traditional Al−12Si braze, in most cases, the interfacial gaps at R/M micro-interfaces could be eliminated finally when small Ti was added, even less than 1%, showing that the developed Al−12Si−xTi active braze ingots could react with Al_2O_3 reinforcement to some extent; moreover, the amount of residual filler decreased with increasing Ti content, showing that the penetration of the Si atoms in the molten brazes into the composite substrate could occur earlier with increasing Ti content.

4) Because the Si atoms in the molten braze foil penetrated into the composite substrate earlier with increasing Ti content, resulting in significant decrease in Si content in the remainder of braze foil, the number of $Ti(AlSi)_3$ phase increased after sessile drop test at 610 °C for 30 min, while the size of $Ti(AlSi)_3$ phase decreased. It should be noted that although small addition of Ti did not affect shear strength and shear fracture surface of the braze ingots significantly, the amount and size of $Ti(AlSi)_3$ phase were sensitive to the Ti content (before brazing) and Si content (after brazing).

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铝基复合材料用 Al−12Si−*x*Ti 系三元活性钎料的制备

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摘　要：为了改善常用钎料对铝基复合材料的润湿性，制备了新型 Al−12Si−*x*Ti 系三元活性钎料。研究表明，所添加的 Ti 以 $Ti(Al_{1-x}Si_x)_3(0\leqslant x\leqslant 0.15)$相存在，但由于钎料的主要组织均为 Al−Si 共晶组织，所以 Al−12Si−*x*Ti 钎料与传统 Al−12Si 钎料具有相似的剪切强度和剪切断口。因此，加入少量 Ti(~ 1%)不会使钎料脆化和硬化；而且 Al−12Si−*x*Ti 钎料带的成分接近共晶成分，所以熔化区间非常相似，为 580~590 °C。对于润湿性的改善，是由于随着钎料中 Ti 含量的增加，Al_2O_3 增强相和钎料金属间的(R/M)界面间隙得到消除的原因。润湿实验后在铝基复合材料表面残留的钎料量也随着钎料含 Ti 量的增加而减少。因此，Ti 对于润湿性的改善可以随着钎料含 Ti 量的增加而较为容易地被重复观察到。此外，$Ti(AlSi)_3$ 的量和尺寸对含 Ti 量(焊接前)和含 Si 量(焊接后)敏感。

关键词：铝基复合材料；钎焊；过渡液相扩散焊；润湿性；钎料

(Edited by YANG Hua)