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Wettability and interfacial reactions for AlN/CuTi and AlN/SnTi systems

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Abstract

Wetting experiments for AlN/CuTi and AlN/SnTi systems were performed through the sessile drop technique in vacuum. Both Cu and Sn do not wet AlN and the final contact angles of the systems are 138° at 1200 °C and 113° at 1000 °C, respectively. In the AlN/CuTi system, the contact angles at the same temperature decrease rapidly when Ti content increases. The final contact angle of the AlN/Cu19.0Ti (the number 19.0 denotes the atomic percentage of Ti) system after holding at 1200 °C for 420 s is 8°. In the AlN/SnTi system, the contact angles decrease rapidly at the beginning of the wetting experiment and stabilized final contact angles are observed. When the Ti content increases from 5 at.% to 15 at.%, the final contact angle decreases from 62° to 60° only. Ti improves the wettability of the AlN/Cu system and the AlN/Sn system through different ways: for the former, by reaction to form a new interface of TiN/CuTi; and for the latter, by diffusion and accumulation of Ti in the interfacial area to form a Ti-rich diffusion layer.

Keywords: Wettability; Reactive wetting; Interfacial reaction; AlN; Alloy

1. Introduction

Aluminium nitride (AlN) is a promising material due to the combination of high strength, high thermal conductivity, high electric resistance and coefficient of thermal expansion close to Si [1]. The characteristics make it an attractive material for many applications, including semiconductor packages, high-frequency acoustic wave devices [2,3].

In some applications, interfaces between AlN and metals are involved, and for this reason many papers deal with the wettability of AlN by liquid metals and alloys. Because of the poor wettability of AlN by metals [4,5], the AlN substrate is difficult to braze. Chemically active brazing elements (M_a), which can improve the wettability between AlN and the alloys, have been used. Ti is the most common one of them. One of the products of the reaction between Ti-containing alloys and AlN is TiN_{1-x} [6–9], which can be wetted by many metals and alloys [10,11], and it is wildly believed that the reaction between Ti and AlN contributes a lot to the improvement of the wettability of the systems. In these cases, the wetting processes between the Ti-containing alloys and AlN substrate are all reactive wetting. Eustathopoulos has presented the ‘‘Reaction Product Control’’ model [12,13], which can explain well the mechanism for Ti to improve the wettability of the systems in these cases. Now the following questions have arisen: the first, is this model applicable for all AlN/Ti-containing alloy systems? The second, are there some other mechanisms for Ti to improve the wettability of the systems? In order to search for the answers to these questions, the wettability and interfacial reactions of AlN/CuTi system and AlN/SnTi system have been investigated and compared with each other in this paper. The effects of the active element Ti on the wettability and reactivity of the systems have been studied. It is anticipated that a better understanding about the mechanisms for Ti to improve the wettability of the systems can be reached.

2. Experimental procedure

In the present study, the raw densified material AlN with a density of 3.30 g/cm^3, which contains about 4 wt.% Y_2O_3 as sintering aid, was used as the ceramic substrate. This material was supplied by Fujian Huaqing Electronic Material Technology Co. Ltd. The grain size of the AlN is in the range of 3–7 μm. The AlN was mechanically processed to the dimension of

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12 mm × 12 mm × 1 mm, and grinded using diamond grinding pans to a roughness of $S_a = 0.46$ μm, and then the samples were cleaned in ethanol for 1200 s using ultrasonic cleaner. The alloys were prepared in situ with Cu powder (99.97 wt.%, 50 μm), Ti powder (99.23 wt.%, 15 μm) and Sn powder (99.9 wt.%, 25 μm), and all of the powders were stored in a vacuum drying oven. The powders were supplied by General Research Institute for Nonferrous Metals in Beijing. The powders were weighed in designed proportion and mixed homogeneously. About 90 mg mixed powders were compacted with a die under a pressure of 600 MPa to form a wafer-shaped compact of $\varphi 5$ mm × 1 mm.

Wetting experiments were performed through the sessile drop technique in an alumina tube in the vacuum of 7×10^{-3} Pa. The furnace consists of a Mo heater around the alumina tube located in a water-cooled stainless steel chamber. The sample was heated in vacuum from room temperature to the designed high temperature at the pace of 9 °C/min, kept holding (if needed), and then furnace-cooled down to the room temperature. The spreading process was photographed by a camera. Each system was repeated for three times. The contact angle (θ) was measured from the drop profile using shape analysis software with an accuracy of ±2°. The selected wetting samples were cut, embedded in a resin and polished. The microstructure of the cross-section of the samples was observed with a Hitachi S-3400 N type Scanning Electron Microscopy (SEM) under the accelerating voltage of 20 KV, and the composition of the cross-section was analyzed with an Oxford Incax-sight 7021 type Energy Dispersive Spectrometer (EDS).

The aqua regia was prepared with hydrochloric acid (12.5 mol/L) and nitric acid (14.5 mol/L), and the volume ratio of the former to the latter was 3:1. To prepare the samples to determine the phase composition by X-ray Diffractometer (XRD), the AlN/Cu10Ti (the number 10 denotes the atomic percentage of Ti, similarly hereinafter) and AlN/Sn10Ti samples were immersed in the aqua regia, and then were taken out as soon as the alloys dissolved completely. A D/max-2200PC type XRD was used to determine the phases of the interlayer at the pace of 6°/min.

3. Results and discussion

3.1. The wettability of the AlN/CuTi system and the AlN/SnTi system

Pure Cu does not wet AlN, and the contact angle θ is 138° in vacuum after holding at 1200 °C for 1800 s. The θ is a little higher than the experimental results reported in Refs. [4,10,14] that θ is 134–135° after holding at 1150 °C for 900 s. There is no appreciable interfacial reaction or adhesion between Cu and AlN after the wetting experiment.

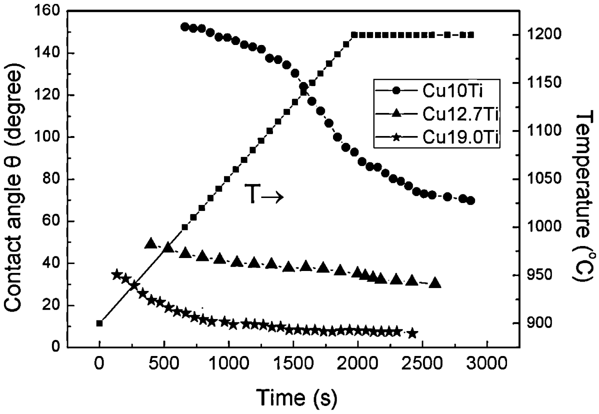


Fig. 1. Contact angle and temperature as functions of heating time for AlN/CuTi systems. Time $t = 0$ is the time when the temperature is 900 °C and the heating-up pace is 9 °C/min.

Fig. 1 shows the contact angles of AlN/CuTi systems when the temperature increases from the melting point of each CuTi alloy to 1200 °C at the pace of 9 °C/min in vacuum. The contact angle of the AlN/CuTi systems decreases with increasing the temperature. The initial contact angle (θ_o) of the AlN/Cu10Ti system is 153°, and θ decreases to 70° after holding at 1200 °C for 900 s. The θ_o of the AlN/Cu12.7Ti system is 50° and the value of θ is 30° after holding at 1200 °C for 600 s. The θ_o and the final contact angle (θ_f) after holding at 1200 °C for 420 s for the AlN/Cu19.0Ti system are 34° and 8°, respectively. Judging from the wetting experimental results, the addition of Ti to Cu can improve the wettability between the Cu and the AlN substrate, especially when the Ti content increases from 10 at.% to 12.7 at.%, the contact angle decreases significantly at the same temperature. This improvement can be attributed to the reaction between the alloy and AlN, which will be discussed below.

Fig. 2 shows the contact angles of the AlN/SnTi systems, when the temperature increases from the melting point of each SnTi alloy to 1000–1200 °C in vacuum. The contact angle of the AlN/Sn system is 113° at 1000 °C. When 1 at.% Ti is added to Sn, the θ_o of the AlN/Sn1Ti system is 112°, θ decreases to 90° at 800 °C and keeps stabilized even the temperature increases to 1000 °C. The relationship between the contact angle and temperature of AlN/Sn2Ti system is similar with that of the AlN/Sn1Ti system, and the θ_o is 140° and θ_f is 77°. The θ_f of the AlN/Sn5Ti, AlN/Sn10Ti and AlN/Sn15Ti systems are 62°, 62° and 60°, respectively, and the temperatures for the three systems to reach a value (64–65°), close to the θ_f, are 1060 °C, 780 °C and 570 °C, respectively.

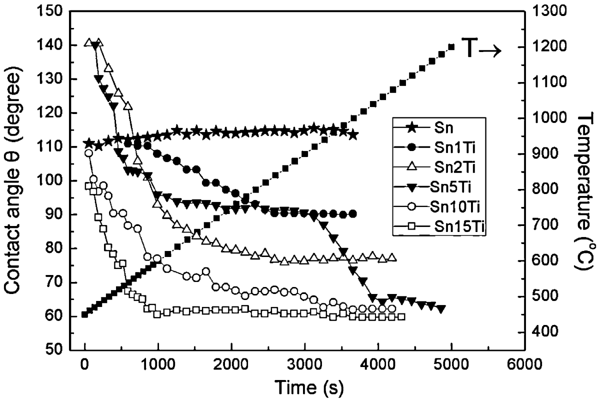


Fig. 2. Contact angle and temperature as functions of heating time for AlN/SnTi systems. Time $t = 0$ is the time when the temperature is 400 °C and the heating-up pace is 9 °C/min.

Judging from the wetting experimental results shown in Figs. 1 and 2, the wetting behavior of the AlN/CuTi systems is quite different from that of the AlN/SnTi systems. In the former systems, the contact angles decrease slowly with increasing the temperature, and no sharp decrease is observed in each system. In the AlN/Cu10Ti and AlN/Cu12.7Ti systems, θs do not reach a stable value even at 1200 °C. In the AlN/SnTi systems, however, the contact angles decrease fast at the beginning of the wetting experiment, and stable final contact angles are observed. When the Ti content increases from 5 at.% to 15 at.%, the final contact angle decreases from 62° to 60° only. These experimental results imply that the mechanism for Ti to improve the wettability of the two kinds of systems is different from each other, which needs to be investigated.

3.2. The reaction in the AlN/CuTi system and the AlN/SnTi system

Let's temporarily ignore the interaction between Ti and Cu in the AlN/CuTi system and the interaction between Ti and Sn in the AlN/SnTi system. Ti contained in the alloys can react with the AlN substrate to form TiN according to the reaction:

$$(\mathrm{Ti}) + \langle \mathrm{AlN} \rangle = (\mathrm{Al}) + \langle \mathrm{TiN} \rangle \quad (1)$$

The notations ⟨⟩ and () designate the solid and the liquid states, respectively, and the product Al dissolves in the alloys. The ΔG_r of this reaction is −43 kJ/mol, calculated by the ΔG_f of each substance at 1200 °C reported in Ref. [15], thus the reaction can occur based on the thermodynamics. However, the Ti element behaves quite differently from each other in the AlN/CuTi system and the AlN/SnTi system.

Fig. 3 shows the micrographs of the interfacial areas of the three typical samples. In the interfacial area of the AlN/Sn sample after holding at 1200 °C for 900 s in vacuum shown in Fig. 3a, some microcracks appear, which indicate poor interfacial bonding between the pure Sn and the AlN substrate. The dark grey particles in the pure Sn are AlN grains, which should be detached from the substrate and were embedded into the soft Sn during preparing the SEM sample. In Fig. 3b, no microcrack exists in the interfacial area of the AlN/Sn5Ti sample after holding at 1200 °C for 900 s in vacuum. Contrasting Fig. 3a with Fig. 3b, the conclusion emerges that the bonding between the Sn5Ti alloy and the AlN substrate is better than that between pure Sn and AlN, and this conclusion has been confirmed by the wetting data in Fig. 2. A reaction layer exists between the CuTi alloy and the AlN substrate shown in Fig. 3c, which presents the microstructure of the AlN/Cu10Ti sample. To determine the composition of the reaction layer in Fig. 3c, the AlN/Cu10Ti sample after holding at 1200 °C for 900 s in vacuum was detected by EDS through line scanning analysis (from A to B) and the results are shown in Fig. 4. Judging from Fig. 4b, both Ti and N are enriched in the reaction layer between the AlN substrate and the alloy, also some Cu exists in the reaction layer, which should diffuse into the reaction layer from the alloy. Fig. 4b also shows that a little Al appears in the reaction layer and in the alloy as well. Since Al is the product of reaction (1), this result suggests that reaction (1) has taken place in the AlN/Cu10Ti system, and a part of the reaction product Al has dissolved in the alloy. With regard to the phase composition of the reaction layer shown in Fig. 4a, it needs to be identified by XRD, which will be discussed below.

Fig. 5 shows the micrograph (back scattering) and the corresponding element mapping (by EDS analysis for Al, Ti and Sn) of a cross-section of AlN/Sn10Ti sample after wetting experiment holding at 1200 °C for 900 s in vacuum. No continuous reaction layer is observed between the Sn10Ti alloy and the AlN substrate, but Ti accumulates in the interfacial area, which should be due to diffusion of Ti into AlN to form a Ti-rich diffusion layer. Two phases including a dark grey one and a light grey one are visible in the Sn10Ti alloy, and the EDS analysis was conducted at point A located at the dark grey phase and point B located at the light grey phase shown in Fig. 5 to determine the composition of the alloy. The results are that point A consists of 41.39 at.% Ti and 58.61 at.% Sn, and point B consists of 100.00% Sn. Thus, the dark grey phase and the light grey phase in the Sn10Ti alloy could be Sn_3Ti_2 and pure Sn, respectively. In order to determine the Ti content of the Ti-rich diffusion layer in the AlN substrate, the compositions of points C, D, E and F shown in Fig. 5 were detected by EDS and

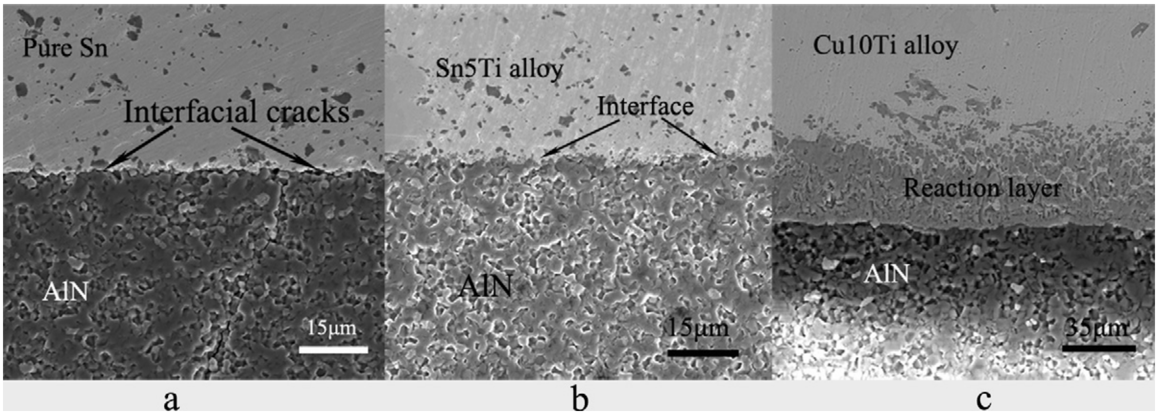
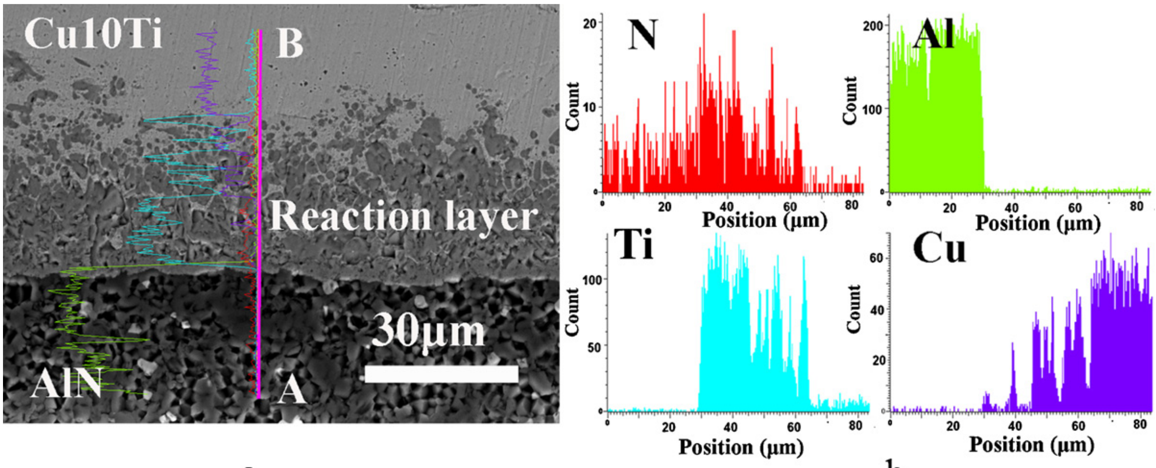


Fig. 3. The micrographs of the interfacial areas of the samples after holding at 1200 °C for 900 s in vacuum. (a) AlN/Sn, (b) AlN/Sn5Ti and (c) AlN/Cu10Ti (secondary electron image).



a

b

Fig. 4. (a) SEM micrograph (back scattering image) and (b) the corresponding element profiles (by EDS line scanning analyzes from A to B for N, Al, Ti, and Cu) of a cross section of AlN/Cu10Ti sample (1200 °C, 900 s, vacuum).

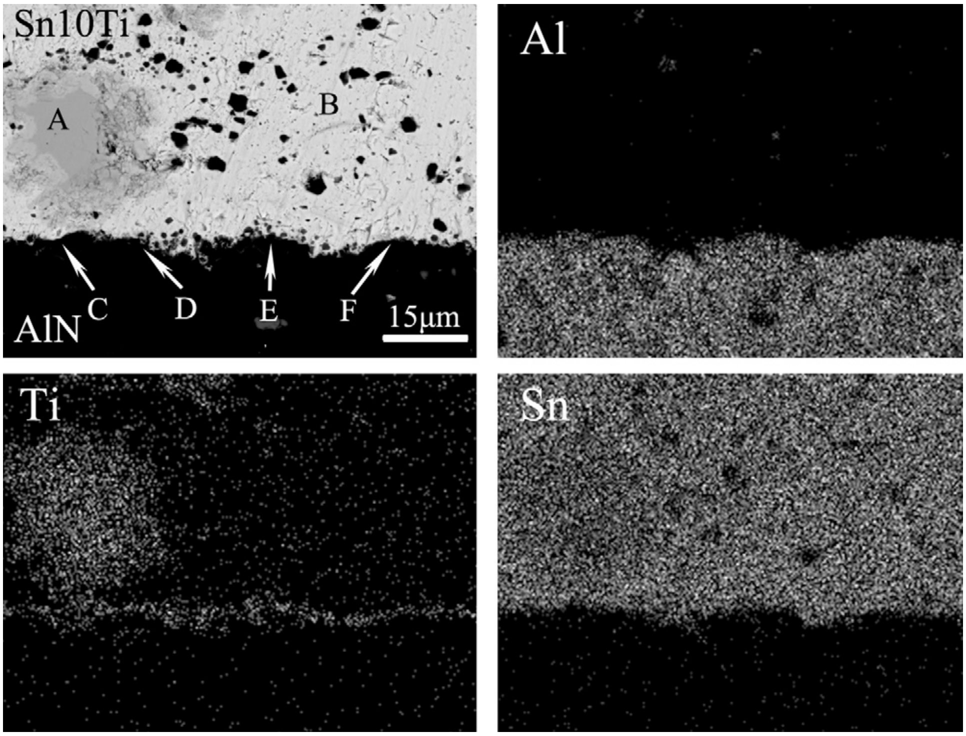


Fig. 5. SEM micrograph (back scattering image) and the corresponding element mapping (by EDS analysis for Al, Ti and Sn) of a cross section of AlN/Sn10Ti sample (1200 °C, 900 s, vacuum).

the results are presented in Table 1. This table shows that the average Ti content is about 8.15 at.% and Sn content is about 5.21 at.%.

In order to identify the phases of the interlayers in AlN/Cu10Ti and AlN/Sn10Ti systems after holding at 1200 °C for 900 s in vacuum, the alloys were carefully dissolved by aqua regia and the interlayers were exposed, and then the samples were analyzed by XRD. The obtained XRD patterns of the interlayers are shown in Fig. 6. This figure indicates that the diffraction peaks of AlN, Y_2O_3 and TiN appear in the XRD pattern of the interlayer in the AlN/Cu10Ti system; however,

Table 1
The results of the EDS spot analyzes in the Ti-rich diffusion layer in AlN substrate for the AlN/Sn10Ti sample (1200 °C, 900 s, vacuum) shown in Fig. 5.

Spot	Compositions (at.%)				
	Ti	Al	Sn	N	Total
C	7.65	55.19	7.15	30.01	100.00
D	7.46	54.64	4.11	33.79	100.00
E	10.07	51.63	3.16	35.14	100.00
F	7.44	50.31	6.40	35.85	100.00
Average	8.15	52.94	5.21	33.70	

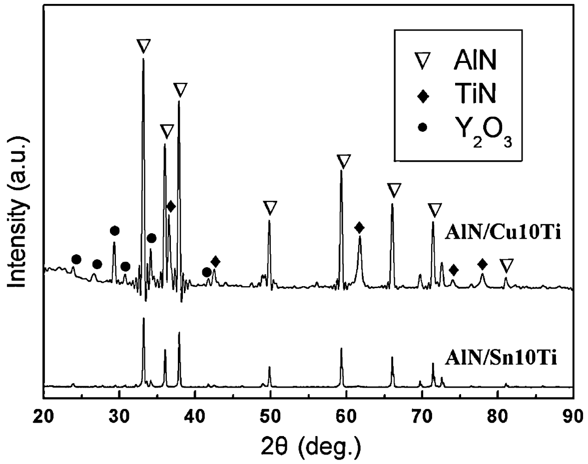


Fig. 6. The XRD patterns of the AlN/Cu10Ti and AlN/Sn10Ti systems (1200 °C, 900 s, vacuum) after removing the alloys (PDF number: TiN: #381420; AlN: #751620; Y_2O_3: #861326).

only the diffraction peaks of AlN and Y_2O_3 are obviously visible in the XRD pattern of the interlayer in the AlN/Sn10Ti system. As TiN is a product of reaction (1), also taking into account that the AlN substrate contains AlN and Y_2O_3 as sintering agent, which does not react with Ti during the wetting experiment [7], the XRD results shown in Fig. 6 confirm that reaction (1) takes place in the AlN/Cu10Ti system rather than in the AlN/Sn10Ti system. So, it can be concluded from Fig. 6 and Fig. 4 that the reaction layer shown in Fig. 4a is composed of TiN as a major phase, some Cu and a little Al. The absence of the diffraction peaks of Cu and Al in the XRD pattern of the interlayer in the AlN/Cu10Ti system shown in Fig. 6 should be due to the dissolution of the metals by aqua regia in the process to prepare the sample. Also, the absence of Ti diffraction peak in the XRD pattern of the interlayer in the AlN/Sn10Ti system shown in Fig. 6 should be due to the same reason. Thus, one can also conclude that the Ti-rich layer in Fig. 5 is not a reaction layer but a Ti-rich diffusion layer.

Comparing the AlN/Sn10Ti system with the AlN/Cu10Ti system, although both alloys contain equal Ti, the wetting behaviors and the interfacial microstructures of the two systems are all quite different from each other. In the former, Ti improves the bonding between Sn and AlN and accumulates in the interfacial area. No obvious reaction layer is observed after the wetting experiment holding at 1200 °C for 900 s in vacuum. In the latter, however, Ti reacts with AlN and a reaction layer is observed under the same condition. These differences indicate that the active element Ti plays different roles in the two systems, and also that the role for Ti to play in the AlN/SnTi systems is not dominated by reaction (1). Since the only difference in the two systems is the different major metal: one is Sn and the other one is Cu, the interaction between Ti and the major metals cannot be neglected. Highly stable intermetallics phases exist in SnTi system [16] but not in CuTi system [17], which suggests that the Ti activity in Sn melt is lower than that in Cu melt. During the dissolution of Ti into the major metals, the interaction may cause energy variation (ΔE), which has been investigated as enthalpy of solution (ΔH_s) by Niessen et al. [18].

In the wetting experiments, the reaction between Ti and AlN in the systems could be divided into the following three steps:

(1) Ti diffuses from the alloy to AlN: ΔE_1.
(2) Ti reacts with AlN at the interface: ΔG_r.
(3) The reaction product Al dissolves into the alloy: ΔE_2.

where ΔE_1 stands for the energy variation when Ti diffuses out of the alloy, and ΔE_2 stands for the energy variation when Al dissolves into the alloy. Since the step (1) could be regarded as the inverse process of the dissolution of Ti into the major metals, one could write that $\Delta E_1 = -\Delta H_s$.

The total energy variation of the reaction between the alloy and AlN in the systems (denoted by ΔG_M) could be expressed as

$$\Delta G_M = \Delta E_1 + \Delta G_r + \Delta E_2 \tag{2}$$

Judging from the data of enthalpy of solution calculated by Niessen et al. in Ref. [18] shown in Table 2, Ti is more likely to dissolve in Sn than Cu, and Al is more likely to dissolve in Cu than Sn. Calculated by Eq. (2), the total energy variation of the reaction between the alloy and AlN in the AlN/CuTi system (denoted by ΔG_{Cu}) and the total energy variation of the reaction between the alloy and AlN in the AlN/SnTi system (denoted by ΔG_{Sn}) are −37 and 45 kJ/mol, respectively. This result may explain the fact that reaction (1) occurs significantly in the AlN/CuTi system but not in the AlN/SnTi system based on thermodynamics. As shown in Table 2, the data of ΔH_s presented in this table are based on the solution at infinite dilution, while the Ti solution during the wetting experiments is not at infinite dilution, thus the calculated values of ΔG_{Cu} (−37 kJ/mol) and ΔG_{Sn} (45 kJ/mol) are for reference only.

Since reaction (1) occurs significantly in the AlN/CuTi system but not in the AlN/SnTi system, the difference of the wetting results between the AlN/CuTi system and the AlN/SnTi system can be explained as follows. In the former, Ti diffuses to and reacts with AlN and a reaction layer mainly composed of TiN forms between the AlN and the CuTi alloy. The other reaction product Al dissolves in the CuTi alloy. Thus the initial solid/liquid interface of the system, which is the interface of AlN/CuTi, is replaced by the interface of TiN/CuTi. Since TiN can be wetted well by CuTi alloy, the wettability of the system is improved. When the content of Ti increases, the reaction layer forms fast and the alloy drop spreads rapidly. The wetting process of the AlN/CuTi system should be controlled by the formation of the TiN reaction layer, since the contact angles decrease slowly with increasing the

Table 2
Enthalpy of solution (ΔH_s, in kJ/mol) at infinite dilution (Ref. [18]).

Solute↓	Solvent	
	Cu	Sn
Ti	−40	−74
Al	−34	14

temperature, and no stable final contact angles have been observed. The experimental results in AlN/CuTi systems agree with the ‘‘Reaction Product Control’’ model presented by Eustanthopoulos in Refs. [12,13]. In the AlN/SnTi system, as shown in Fig. 2, the contact angles decrease fast only at the beginning of the wetting experiments and then reduce slightly, which suggests that the formation of the Ti-rich diffusion layer should control the wetting process. If the formation of the Ti-rich layer in Fig. 5 is dominated by reaction (1), the Ti-rich layer should be mainly composed of TiN, and the other reaction product Al should dissolve in the SnTi alloy. Thus, the concentration of Ti in the Ti-rich layer should be close to that of N and much higher than that of Al, and also Al should be detected in the SnTi alloy. However, the experimental results of the composition of the Ti-rich layer shown in Table 1 are on the contrary. The concentration of Ti (8.15 at.%) is lower than that of N (33.70 at.%) and that of Al (52.94 at.%). Moreover no Al was detected in the SnTi alloy. For comparison purpose, it should be mentioned that Al has been found in the reaction layer and the alloy in the AlN/Cu10Ti system as shown in Fig. 4, also as reported in Ref. [19], some Al is found in the CuSiTi alloy after the wetting experiment of AlN/CuSiTi system, where a reaction layer mainly composed of TiN forms. In brief, the experimental results suggest that the Ti-rich layer in Fig. 5 is not a reaction layer produced by reaction (1) but a Ti-rich diffusion layer, and its formation should dominate the wetting process. The experimental results presented in Figs. 2 and 5 suggest that the formation of the Ti-rich layer in the interfacial area contributes to the improvement of the wettability of the system. When a Ti-rich layer forms in the interfacial area, the contact angle of the system should become an approximately stabilized value. Obviously, the more the Ti content in the alloy, the easer the formation of the Ti-rich layer by Ti diffusion and accumulation, and consequently the lower the temperature at which the approximately stabilized contact angle of the system is obtained. This is in accordance with the experimental results shown in Fig. 2. For example, the temperatures to obtain the approximately stabilized contact angle for the AlN/Sn5Ti, AlN/Sn10Ti and AlN/Sn15Ti systems are 1150 °C, 950 °C and 600 °C, respectively. These experimental results indicate that the ‘‘Reaction Product Control’’ model in Refs. [12,13] is not applicable for AlN/SnTi system.

4. Conclusion

Wetting experiments for AlN/CuTi and AlN/SnTi systems were performed through the sessile drop technique in vacuum, and the following conclusions emerge from the present data:

(1) Both Cu and Sn do not wet AlN and the final contact angles of the systems are 138° at 1200 °C and 113° at 1000 °C, respectively.
(2) The addition of Ti can improve the wettability for both AlN/Cu and AlN/Sn systems. But the wetting behaviors of the two systems are quite different from each other. In the AlN/CuTi system, the contact angles at the same temperature decrease rapidly when the Ti content increases. The final contact angle of the AlN/Cu19.0Ti system after holding at 1200 °C for 420 s is 8°. In the AlN/SnTi system, within the tested range of Ti content 1–15 at.%, the lowest contact angle is 60°. When the Ti content increases from 5 at.% to 15 at.%, the final contact angle decreases only from 62° to 60°.
(3) Ti improves the wettability of the AlN/Cu system and the AlN/Sn system through different ways: for the former, by the reaction between Ti and AlN to take place, leading to the formation of a new interface of TiN/CuTi; and for the latter, by diffusion and accumulation of Ti in the interfacial area, leading to the formation of a Ti-rich diffusion layer. The ‘‘Reaction Product Control’’ model presented in Refs. [12,13] is applicable for AlN/CuTi system, but not for AlN/SnTi system.

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