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

Chuanbao Wang [a], Shujie Li [a,*], Shuzhi Li [b], Ting Zhang [a], Chao Ma [a]

[a] School of Materials Science and Engineering, Beijing University of Aeronautics, and Astronautics, Beijing 100191, PR China
[b] Beijing Institute of Automatic Control Equipment, Beijing 100074, PR China

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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

* Corresponding author.
E-mail address: shujieli@buaa.edu.cn (S. Li).

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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 φ5 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 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.

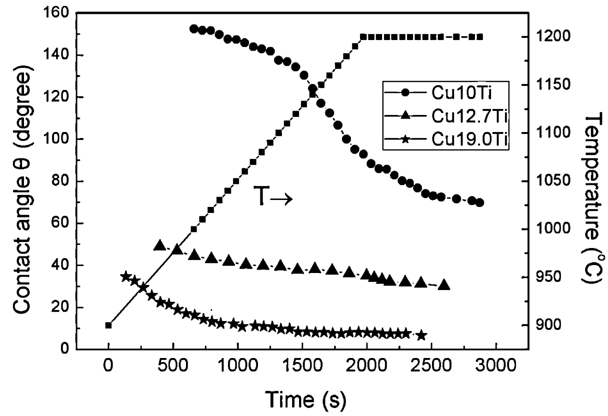

Fig. 1. Contact angle and temperature as functions of heating time for AlN/CuTi systems. Time t = 0 is the time when the temperature is 900 °C and the heating-up pace is 9 °C/min.

Fig. 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.

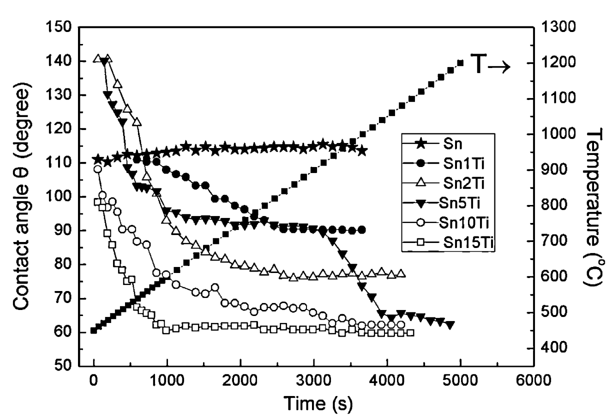

Fig. 2. Contact angle and temperature as functions of heating time for AlN/SnTi systems. Time t = 0 is the time when the temperature is 400 °C and the heating-up pace is 9 °C/min.