



## Letter

Wetting behavior of Al alloys on a TiH<sub>2</sub> substrateDawu Li<sup>a</sup>, Jie Li<sup>a</sup>, Xiaoming Zhang<sup>b</sup>, Ting Sun<sup>a,\*</sup>, Guangchun Yao<sup>b</sup><sup>a</sup> Department of Chemistry, Northeast University, Shenyang 110004, China<sup>b</sup> School of Materials and Metallurgy, Northeast University, Shenyang 110004, China

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## ABSTRACT

The wetting behavior of Al–Ge alloys on TiH<sub>2</sub> substrates was investigated by an improved sessile drop method under high vacuum and in a temperature range of 773–818 K. Results indicate that the equilibrium contact angles of Al–Ge/TiH<sub>2</sub> increase linearly with temperature according to the following formula:  $\theta = 0.2882T - 85.04$ , and decrease linearly as the Ge content increases from 25.2% to 36.2% according to the formula:  $\theta = 214 - 200\text{Ge}$  (wt.%). The worst wetting behavior was obtained for a pure Al/TiH<sub>2</sub> system at its foaming temperature (973 K). TiH<sub>2</sub> particles were prone to aggregate and were thus difficult to disperse. This could be one of the reasons for closed-cell aluminum foam products having non-uniform pores.

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## 1. Introduction

Closed-cell aluminum foams (CAF) are ultralight materials which are used as functional materials for sound absorption, energy absorption and fire resistance, etc. [1–3]. The properties of CAF depend on many morphological features such as the pore size distribution and cell wall curvature defects [4]. CAF with fine, uniform cell structures are of interest to researchers [5,6]. Although several methods to fabricate metal foams exist, it is still difficult to obtain a homogenous structure with uniform properties [7]. Most studies have focused on the thermodynamic instability of gas expansion in the melt as non-uniform pores exist because the bubble usually undergoes growth, amalgamation and disintegration in the molten aluminum metal [8–10]. On the other hand, it has been reported that the presence of particles in the molten metal helps to stabilize the foam by allowing for accumulation on cell walls [7]. Because the foaming process results in an unstable system that consists of gas, liquid and solid phases [9,11], the particle wetting behavior is considered to have an important influence on the foaming behavior of particle suspensions [12,13]. Partially wetted particles accumulate at bubble surfaces where they act as a steric (mechanical) barrier against coalescence. Foams that incorporate finely divided solid particles are frequently encountered during the processing of many industrial products such as foods or chemical products [13]. Recently, it has been shown that solid particles with specific surface

properties can be used to generate ultrastable particle-stabilized foams [14]. In theoretical models by Kaptay [15], the stabilizing effect of these particles on the liquid aluminum foam is due to capillary effects between the melt and the particles and this prevents the two liquid gas interfaces in a foam cell wall from touching and the two adjacent cells from coalescing. Titanium hydride (TiH<sub>2</sub>) particles are usually used as a blowing agent because they release gas and expand liquid aluminum during the preparation of aluminum foams [16]. The dispersion of TiH<sub>2</sub> is one of the most important factors that influence the preparation of uniform CAF. The quality of the produced aluminum foams is influenced by the foaming agent particle distribution. Therefore, understanding the wetting behavior of aluminum on TiH<sub>2</sub> is crucial to obtain stable foams during the production of CAF.

However, few reports exist on the wettability of molten aluminum on TiH<sub>2</sub>. The difficulty in wetting research is mainly that the maximum endothermic peak of TiH<sub>2</sub> (873 K) [8,17] is significantly lower than the melting point of aluminum (933 K) and TiH<sub>2</sub> usually decomposes at the melting temperature of aluminum. It is crucial to choose an aluminum alloy with a high aluminum content to make the experiment more representative. To reduce the impact of TiH<sub>2</sub> decomposition as much as possible, a low melting Al–Ge alloy [18] was selected to replace Al.

In this study, the wetting behavior of TiH<sub>2</sub> by a molten Al–Ge alloy in a temperature range from 773 K to 818 K using an improved sessile drop device was investigated. The effects of temperature and Ge content (wt.%) on the wettability were, therefore, investigated.

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## 2. Experimental

High purity aluminum (99.99%) and blocks of TiH<sub>2</sub> (99.99%) were obtained from Fushun Aluminum Company Limited and Germanium (99.99%) was obtained from Qingdao Xinyan Non-ferrous Metals Company Limited. Al–Ge alloys were prepared by the high-frequency vacuum induction melting method at 1073 K for 2 h and the Ge content of the samples was determined by ultraviolet–visible spectrophotometry at 512 nm [19]. For the wetting experiments, Al–Ge samples were cleaned in a 10 wt.% NaOH solution to remove the surface oxide film before the samples were immersed in acetone and cleaned ultrasonically. An Al–Ge sample (0.15–0.25 g) and a magnet were then placed into a glass tube that was located outside the chamber. The glass tube was connected to a corundum dropping tube (Al<sub>2</sub>O<sub>3</sub> 99.6 wt.%) with a  $d = 2$  mm hole at its bottom. The Al–Ge sample could thus be transferred through this hole into the chamber and then dropped onto the substrate when the desired test temperature was reached. The TiH<sub>2</sub> substrate was placed in a stainless steel chamber and adjusted to be horizontal, as shown in Fig. 1. The system was evacuated ( $2 \times 10^{-3}$  Pa) and purged with high purity argon (99.9999%, IMR, Shenyang) once through the chamber. The furnace was heated to the desired temperature using a temperature program controller with a Pt–Rh/Pt thermocouple. Subsequently, the Al–Ge pieces were pushed into a corundum pipe using a magnet and were held at that temperature for 15 min.

As soon as the Al–Ge alloy drop was formed on the TiH<sub>2</sub> substrate, a photograph was taken with a digital camera (7 million pixels, model ESE-220c, Shenyang) and defined as the drop profile at zero time. Subsequent photos were taken at specific time intervals. The captured photographs were analyzed by the method given in previous reports [20]. To analyze the interfacial characteristics of Al–Ge/TiH<sub>2</sub>, solidified samples were vertically sectioned to prepare metallographic specimens. The interfacial microstructures were examined using a scanning electron microscope (Shimadzu SSX-550, Japan) with energy dispersive X-ray microanalysis (EDX) capability.

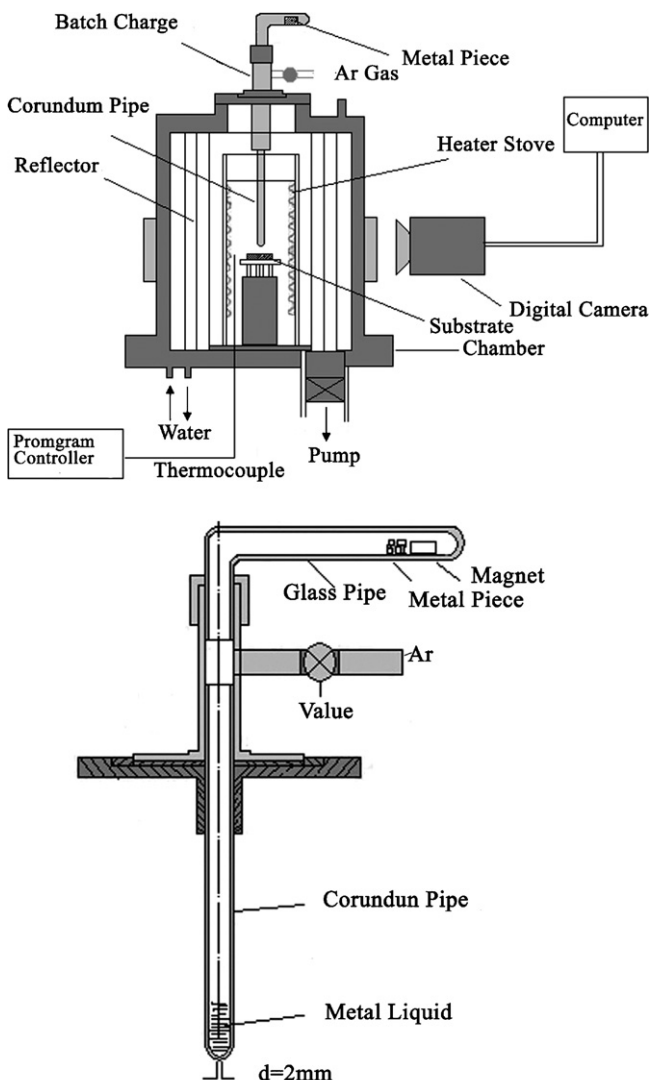


Fig. 1. Schematic of the experimental apparatus.

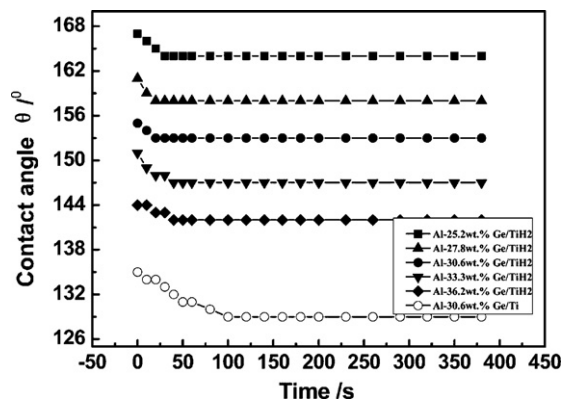


Fig. 2. Variation in contact angles with time for Al–Ge/TiH<sub>2</sub> with different Ge content at 818 K.

## 3. Results and discussion

### 3.1. The effect of Ge content on the contact angle

The Ge content of the Al–Ge alloys that were produced by high-frequency vacuum induction was examined. The average Ge content (wt.%) values ( $n = 3$ ) were found to be 25.2, 27.8, 30.6, 33.3, 36.2, respectively. Fig. 2 shows changes in the contact angles of the molten Al–Ge alloys on the TiH<sub>2</sub> and Ti substrates for the alloys with different Ge content. These contact angles quickly equilibrate and do not show any apparent changes after 6 min. It is worth noting that the contact angles of the Al–Ge alloys on TiH<sub>2</sub> substrates are largely influenced by the Ge content. By increasing the Ge content (wt.%) from 25.2 to 36.2, the initial contact angle decreases from 167° to 144° and the equilibrium contact angle ( $\theta_{eq}$ ) is also reduced from 164° to 142°. The contact angle of the Al–Ge alloy on Ti is lower than its contact angle on a TiH<sub>2</sub> substrate. All these curves describe similar behavior, which can be classified in two stages. The first stage is a rapid decrease in the contact angle with processing time. The second stage is characterized by the contact angle gradually stabilizing, which results from an energetic equilibrium constraint on the surface. This behavior indicates that better wettability is gained from higher Ge content. These results are consistent with the previous report by Taranets [18] for an Al–Ge/AlN system.

The equilibrium contact angles, as shown in Fig. 2, were fit to a linear equation. The relationship between the Ge content (wt.%) and the contact angle ( $\theta_{eq}$ ) is given in Eq. (1). This phenomenon (Eq. (1)) indicates that the investigated Al–Ge alloy has better wetting behavior at a higher Ge content.

$$\theta_{eq} = 214 - 200Ge \text{ (wt.%) (range 25.2 – 36.2\%)} \quad R_1 = 0.9985 \quad (1)$$

### 3.2. The effect of temperature on the contact angle

The equilibrium contact angles for the Al–Ge alloy on TiH<sub>2</sub> that were obtained between 773 K and 818 K are shown in Fig. 3. The contact angle clearly depends on the temperature. By increasing the temperature from 773 K to 818 K, the initial contact angles increased from 140° to 155° and the equilibrium contact angle ( $\theta_{eq}$ ) also changed from 137° to 153°. The equilibrium contact angle as shown in Fig. 3 was also fit to the linear equation in Eq. (2). This indicates that higher temperatures lead to worse wettability.

$$\theta_{eq} = 0.2882T - 85.04 \text{ (temperature range 773 – 818 K)} \quad R_2 = 0.9984 \quad (2)$$

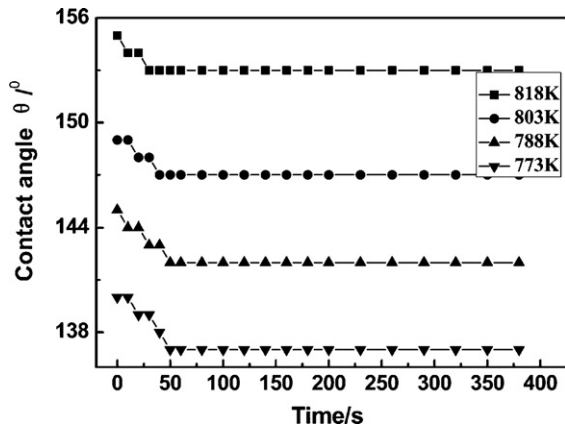


Fig. 3. Variation in contact angles for various temperatures at 30.6% Ge content in Al-Ge/TiH<sub>2</sub>.

### 3.3. Surface analysis

Surface analysis showed that no new compounds were generated at the interface and no new phases were found other than Al, Ge and Ti using the experimental conditions described previously.

### 3.4. Discussion on the effect of the wetting behavior of Al/TiH<sub>2</sub> on aluminum foam preparation

Eqs. (1) and (2) exhibit good linear relationships with a change in the Ge content from 25.2% to 36.2% and with a change in temperature from 773 K to 818 K. The equilibrium contact angle ( $\theta_{eq}$ ) decreases constantly with an increase in the Ge content and an increase in the temperature. The worst wetting behavior for the Al-Ge/TiH<sub>2</sub> system is observed at higher temperature and for pure aluminum. Therefore, the contact angles of Al/TiH<sub>2</sub> show worse wetting behavior at the foaming temperature (973 K).

Because wettability has a great influence on the dispersion of particles in a liquid [13–16], it is not difficult to recognize that particles of TiH<sub>2</sub> are prone to aggregate when adding TiH<sub>2</sub> particles to Al melts during the foaming process. They, therefore, disperse with difficulty because of the worse wettability of the Al/TiH<sub>2</sub> system. This leads to the non-uniform distribution of the foaming agent particles (TiH<sub>2</sub>). Therefore, a high intensity mechanical stirring process must be employed to disperse TiH<sub>2</sub> uniformly. This adds to the

complexity of the experimental process during the CAF preparation process.

## 4. Conclusions

Wetting results for Al-Ge alloys on a TiH<sub>2</sub> substrate show that the equilibrium contact angle  $\theta_{eq}$  is higher at higher temperature according to the formula:  $\theta_{eq} = 0.2882T - 85.04$ . The contact angle decreases as the Ge content (wt.%) increases according to the formula:  $\theta_{eq} = 214 - 200Ge$  (wt.%). These changes indicate that the Al/TiH<sub>2</sub> system seems to undergo worse wetting at the foaming temperature of the CAF. This may be one of the reasons that final CAF products have non-uniform pores.

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