Research Article
Contact and Infiltration Behavior of Al Melt and Al-B_4C Composite

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Received 25 January 2022; Revised 28 May 2022; Accepted 31 May 2022; Published 29 June 2022

Academic Editor: Dimitrios E. Manolakos

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In the present study, the authors used sessile drop method to simulate the contacting and infiltration behavior between Al melt and Al–B_4C composite. In order to track the penetration distance of the metal melt infiltrate into the Al-B_4C composite matrix when the infiltration is not obvious, element Si was added into the metal melt as the tracer agent. The Al-10%Si melt was dropped onto the Al-B_4C composite substrate at 913K. The changes of droplet bottom size, height, and contact angle during the spreading and penetration with Al-B_4C composite substrate surface are investigated by infiltration experiment equipment. The process of the droplet infiltrating into the Al-B_4C composite can be divided into two stages: (a) a slow infiltration stage that the contact angle changes slowly; (b) a quick infiltration stage with the rapidly-changed contact angle. The content of B_4C and the pores in the composite plays an important role on the infiltration of the droplet. The higher the density, the easier the infiltration proceeds.

1. Introduction

Metal matrix composites (MMCs) reinforced with ceramic phase have emerged as an important class of materials for structural, wear, thermal, transportation, and toughness, because of their excellent properties compared with the corresponding monolithic alloy [1]. Among them, three-layered composite materials have been widely used in many industrial fields because of their excellent physical, chemical, and mechanical properties that cannot be obtained from a single material [2–4]. The metal-cermet three-layered composite combines the advantages of both metal matrix composites and the three-layered composite materials [5, 6]. It is an ideal material in the areas of light protection and bulletproof armor for which ballistic performance is an important index.

Boron carbide (B_4C) is one of the most commonly used reinforced materials [7–9]. An outstanding mechanical property of B_4C is its hardness, which is only lower than that of diamond and c-BN [10]. This specific property comes along with other attractive properties such as high impact and wear resistance, low density, high melting point, and excellent resistance to chemical agents, as well as high capability for neutron absorption; many researchers use B_4C as an ideal reinforcement phase [11, 12]. However, its extreme sensitivity to brittle fracture (K_{IC} = 3.7 MPa m^{1/2} [13]) highly limits its use in industrial applications. Aluminum and aluminum alloys have the properties of low density and excellent toughness [14]. Boron carbide appears to be an interesting strengthening agent to compensate the deficiency of aluminum [15, 16].

Powder metallurgy is the most widely-used method for the fabrication of B_4C–Al composites [17, 18]. Component proportion of each component can be controlled easily. Complex shapes of product can be achieved without much mechanical working. Among them, powder rolling is a high-yield and efficient method [19]. The authors present a new-layered composite fabricated by semicontinuous casting and powder rolling process. The layered composite possess a sandwich structure, the outer layers are made of Al alloy, and the inner layer is B_4C reinforced composite.

However, the combination process between layers is not clear. This paper is interested in the manner for the contact and infiltration between Al alloy melt and B_4C–Al composite. The B_4C and Al powder was hot pressed and sintered by powder metallurgy. Sessile drop technique was invited to...
simulate the contact and infiltration process between layers, as well as the microstructure of the infiltration area and the changing of the droplet.

2. Experimental Study

Infiltration process of the metal melt in contact with the Al–B₄C composite matrix is uncertain, in order to track the penetration distance of the metal melt infiltrate into the Al–B₄C composite matrix when the infiltration is not obvious; alloy elements were added into the metal melt as the tracer agent. Common alloying elements in Al alloys include Mg, Si, and Cu. Metallic elements usually react with Al to form brittle intermetallic compounds, which affects the observation of interface behavior. The surface tension of the alloy droplets determines the infiltration process between the alloy droplets and the Al–B₄C composite matrix, so the selected alloy elements need to have minimum influence on the surface tension of pure Al melt. As shown in Figure 1, it shows the effect on surface tension of alloying elements in molten Al (99.99%).

It can be found from Figure 1 that the surface tension of molten Al remained the same after the addition of alloying element Si without any serious reaction. Therefore, Al–10%Si alloy is selected to investigate the interface formation process between Al–B₄C composite matrix and Al–10% Si alloy.

The B₄C-Al composite substrates were prepared by mechanical alloying and hot-pressing process. Analytical pure fine Al powder and B₄C(d72 μm) were used; the mass ratio of boron carbide particle and matrix Al are 37.5% and 62.5%, which have been proven to possess a better mechanical property. The mixed powders were pretreated in SFM-1 planetary ball miller for 1 h: sun-disk rotation speed 300 rpm and planetary-disk rotation speed 510 rpm. The ball-to-powder weight ratio was 1:1. The direction of rotation changed every half hour to ensure that they can be mixed thoroughly.

The milled powders were enclosed in an alloy steel die (inner size Ø70 mm × 80 mm, outer size Ø80 mm × 80 mm) and cold compacted under 40 MPa to prepare the nominal dimension of Ø 70 mm × 4 mm composite.

Six specimens (referred as MMCs-1 to MMCs-3) were hot pressed under different pressures at 823 K for 2 hours and followed by furnace cooling.

The mass and volume of the specimens were measured by the Archimedes method, and the relative densities are shown in Table 1.

3. The Contact Experiment

The contact experiment was investigated by infiltration experiment performed in the vacuum at isothermal environment using the sessile drop method. The sessile drop technique is a standard method used in wettability studies and infiltration and wetting observation.

The composite substrate was preplaced horizontally in the furnace while the Al-Si alloy was stored in a foldable metallic tube outside the chamber (shown in Figure 2).

Before the experiment, the composites were cut by electrodischarge machining (EDM) into nominal dimension of 20 mm × 20 mm × 4 mm, and the upper surfaces were polished from 800 to 1200 grit emery paper. Both the polished substrate and the Al–10%Si alloy were carefully ultrasonically cleaned in acetone before placed into the chamber.

The substrate was heated in a high vacuum (approx. 9.0·10⁻⁴ Pa) to 913 K, holding for 10 min. Then, the Al–10% Si alloy was placed into the bottom of an alumina tube (in 99.7 wt.% purity) which is placed in the vacuum chamber just above the substrate and heated for 60 s at 913K as well. Then, the pressure of the chamber is reduced, and the melting alloy was extruded through a small orifice, rested on the substrate under the effect of atmospheric pressure. During the melting extruded from the orifice, the shape of the droplet changes a lot; therefore, the oxide film covering on the Al–10%Si surface was mechanically broken.

The start of experiment was defined when the liquid contacted the substrate surface. The photos were taken at a maximum speed of 1 frame per second. The experiment retained for 30 min, followed by furnace cooling. In addition to the infiltration data, the method provided quantitative information on wetting, thus allowing the spreading and infiltration rates to be measured in the same experiment.

After the experiment, the captured drop profiles were analyzed by a drop-analysis program to calculate the contact angles, and the infiltration rate was calculated from the photography. The cross-section microstructures of the substrates were observed using a scanning electron microscope (SEM, Carl Zeiss SUPRA55, Germany) equipped with an energy dispersive spectrometer (EDS).

![Figure 1: Effect of alloy elements on the surface tension of molten Al(99.99%).](image)

| Table 1: Parameters of specimens with different pressure. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| Composite (No.) | Pressure (MPa)  | Mass (g)        | Volume (cm³)    | Density (g/cm³) |
| MMCs-1          | 120             | 4.2788          | 1.7776          | 2.4071          | 0.9139          |
| MMCs-2          | 80              | 4.9947          | 1.6290          | 2.4522          | 0.9311          |
| MMCs-3          | 40              | 4.9598          | 1.7027          | 2.3256          | 0.8830          |
4. Results and Discussion

Figure 3 shows the contact process of Al–10%Si alloy and Al–B$_4$C composite substrate at 913 K. After contact, the alloy droplet starts to infiltrate into the substrate, and the volume of the droplet becomes smaller. In the meantime, the substrate around the alloy droplet upheaves as highlighted by the white arrows in Figure 2. In addition, the infiltration rate of the MMCs-2 is faster than that of MMCs-1 and MMCs-3.

After the droplet contact with the substrate, the edge of droplet contacts several B$_4$C particles (as shown in Figure 4). According to Young’s equation [20],

$$W = \alpha W'_a - \beta W'_c = \alpha \cdot \gamma_{lv} (1 + \cos \theta) - \beta \cdot \gamma_{lv} (1 + \cos \theta'),$$

where $W$ is the adhesion work of the Al–10%Si alloy droplet to the Al–B$_4$C composite substrate, $W'_a$ and $W'_c$ are the adhesion work of the droplet to Al and B$_4$C respectively, $\alpha$ and $\beta$ are the volume fractions of Al and B$_4$C in the substrate respectively, $\theta$ and $\theta'$ are the contact angles of the droplet to Al and B$_4$C respectively. The volume fractions of boron carbide particle and matrix Al are 39.3% and 60.7%. The contact angle of Al–10%Si melt to Pure Al is near 0, and that of Al–10%Si melt to B$_4$C is more than 100.

From the equation, the adhesion work of droplet to Al is much higher than that of droplet to B$_4$C; the droplet should spread on the substrate. However, the substrate is not a totally smooth surface even after polishing due to the hardness difference between Al matrix and B$_4$C particle. As we all know, the Al matrix is much easier to be polished than B$_4$C particle. Figure 5 shows the schematic diagram of Al–10%Si alloy melt droplets contact the surface with different roughness. The contact angle of the metal droplets contact with the smooth surface is smaller than that of the rough surface. The rough surface of Al–B$_4$C composite matrix will hinder the spreading of the metal droplets on it.

The changing of the contact angle after contact (the time-dependent change) is shown in Figure 6. The contact process can be roughly divided into two stages: (a) a slow infiltration stage in this stage that the contact angle slowly changes; (b) a quick infiltration stage. The curves of MMCs-1 and MMCs-2 are superposed. The contact angle of MMCs-1 and MMCs-3 decreases to zero in about 140 seconds and 145 seconds after contact. The contact angles decrease with the infiltration proceeding. Unlike MMCs-1 and MMCs-3, the contact angle of MMCs-2 rapidly decreases in the first 5 seconds, which means a quick spread happened. After the rapidly decrease, the contact angle of MMCs-2 maintains smaller than that of the other two. The contact angle of MMCs-2 decreases to zero in about 95 seconds after contact.

The volume change of the droplet is shown in Figure 7. The bottom size of MMCs-2 is bigger than the other two. The bottom size decreases in a slow rate at first, after a hemispheric droplet left on the substrate, the bottom size begins to decrease rapidly. There is no spread process found in droplet bottom changing curves, except a little increasing during 10 s to 30 s after contact. The height of the droplet decreased under an almost constant rate.

The Al–B$_4$C composite matrix with different densities shows different infiltration modes. The difference of relative density makes the porosity of the samples different. Sample MMCs-2 with the highest relative density has a lower porosity, and sample MMCs-3 with the lower relative density has a higher porosity. When the alloy droplet drops onto the surface of Al–B$_4$C composite matrix, the droplet contacting with the metal part of Al–B$_4$C composite surface spreads out, and the droplet above the pores penetrates through the pores. For the samples MMCs-3 and MMCs-2 with low porosity, the Si is more inclined to infiltrate into the matrix metal part rather than penetrating through the pores. The diffusion of Si causes the alloying of the metal matrix to be in contact with the alloy droplet and the increase of liquidus. The molten matrix alloy and the alloy droplet flow together at the same time, thus accelerating the further diffusion of Si into Al–B$_4$C matrix. Therefore, the diameter-time curves of the samples MMCs-1 and MMCs-2 decrease slowly at beginning and then rapidly decrease, because the alloy droplets in contact with the Al of Al–B$_4$C composite matrix tend to spread out, so their infiltration mode is similar to Figure 8(b). The bottom diameter of the droplet decreased at almost the same rate after contact with sample MMCs-3 (the lowest relative density). Lower relative density leads to higher porosity; liquid alloy under the effect of capillary force directly penetrates into the pores of the Al–B$_4$C composite substrate, because the high porosity brought larger surface roughness, the trend of alloy droplet
Metal droplet contact smooth surfaces

Metal droplet contact rough surfaces

Figure 3: The contact process of droplet and substrate composite at 913K.

Figure 4: Top view of the droplet contacting with the substrate.

Figure 5: Al droplet contact with different surface of Al–B\textsubscript{4}C composite matrix.

Figure 6: The contact angles changing after droplet contact with the substrate.
Figure 7: The volume change after the droplet contact with the substrate.

Figure 8: The schematic diagram (a) of alloy droplet infiltrates (b) into Al-B₄C composite substrate.

Figure 9: The cross-section microstructures of the substrates from MMCs-1 (a) Cross-section microstructure of MMCs-1; (b) microstructure of remelted area of MMCs-1.
spreading outward is far less than the other two samples; therefore, the infiltration pattern is similar to Figure 8(a).

The microstructure of MMCs-1 was taken as the typical example. Figure 9 presents the microstructure of cross section of MMCs-1. It clearly shows that the boron carbide near the surface is replaced by the melting of the substrate. At the same time, a remelted area was left near the drop-substrate interface.

The diffusion of the Si element into the substrate decreases the liquidus temperature of the contacting area. The substrate surface begins to melt when its liquidus temperature decreases to 913 K. The melt of the Al matrix surface helps the further diffusion of the Si element. The melted area expands after the melt of the matrix surface, and the volume of the melted increases. This is the reason of the upheaved on the substrate surface. As the bottom size becomes smaller during the diffusion after half of the droplet diffused into the substrate, the diffusion rate of Si element from the droplet decreased. The section of melted area far away from the droplet lacked of Si element and gradually solidified during the further diffusion. The Si atom of the melted area under the droplet is still enough to diffuse deeper. The remelt of the substrate is shown in Figure 10.

5. Conclusion

For the layered composites, the liquid metal combined with the ceramic B4C particle reinforced Al matrix composite layer by wetting on the surface and infiltrate into the porous of the composite. The process of the liquid metal combined with the composite can be divided into two stages: (a) a slow infiltration stage and contact angle changed slowly; (b) a quick infiltration stage and the contact angle changed rapidly.

The content and distribution of the B4C particles in the composite play an important role to influence the infiltration of the droplet. In the meantime, the substrate roughness and the distribution of the pores also influence the infiltration of the droplet. The infiltration of the droplet accelerated the element diffusion and the remelt of the substrate. The raise of the substrate surface is because that the substrate is remelted by the droplet.

The density of the substrate is the most significant factor on the contact and infiltration process. When the pressure is 80 MPa, the relative density of the substrate is 0.9706 and the droplet infiltration time is shorter than the other two specimens.

The results show that Al melt can directly contact Al–B4C composite matrix if a semicontinuous casting method is designed, and the density of Al–B4C composite matrix is lower than that of Al–B4C composite matrix used in this paper. According to the results of this paper, it can be inferred that Al–B4C composite matrix can form a strong metallurgical interface with Al melt, and its infiltration mode is a direct infiltration of metal melt into Al–B4C composite matrix. The results of this paper provide a theoretical basis for the preparation of Al/(Al–B4C)/Al layered composite slab by semicontinuous casting and hot rolling.

Data Availability

The data used to support the findings of this study are included within the article and are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

This work was supported by the “Scientific research fund project of Education Department of Liaoning Province” (No. JL202010); PhD initiation research fund project of Dalian Ocean University (HDYJ202109); National Natural Science Foundation of China (Grant no. 51902039); Dalian High-level Talents Innovation Support Plan (Grant no. 2020RQ127).

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