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# Influences of surface polarity and Pd ion implantation on the wettability of Al-12Si(-2 Mg)/SiC systems



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#### HIGHLIGHTS

- The Al-alloy/C-terminated SiC have a better wettability.
- The 2% Mg can improve the wettability of Al-12Si/SiC system.
- The wettability of Al-12Si/SiC increased with increasing Pd-implantation dose.
- The wettability of Al-12Si-2Mg/SiC decreased with the increasing of Pd-implantation dose.

#### ARTICLE INFO

Article history:
Available online 22 February 2018

Keywords: Implantation Interfaces SiC Wettability

#### ABSTRACT

N-type 6H-SiC (0001) single crystals were implanted with 20 keV Pd ions at three doses of  $5\times10^{15}$ ,  $5\times10^{16}$  and  $5\times10^{17}$  ions/cm² at room temperature. The wetting of SiC single crystal by molten Al-12Si and Al-12Si-2Mg were performed by using the sessile drop method in a high vacuum at 1323 K. The influences of surface polarity and Pd ion implantation on the wettability of Al-12Si(-2 Mg)/SiC systems were investigated. The experimental results showed that the equilibrium contact angles of molten Al-12Si and Al-12Si-2Mg on the C-terminated SiC were respectively lower than those on the Siterminated SiC, which can be related to the formation of Al<sub>4</sub>C<sub>3</sub> at the interface. The wettability of Al-12Si/C(Si)-terminated SiC systems was improved with the 2 wt% Mg addition due to the decrease of the solid-liquid surface energy ( $\sigma_{SL}$ ) originated from the enhancement of interfacial interactions. Moreover, the equilibrium contact angles of Al-12Si/C(Si)-terminated SiC systems decreased more or less with the Pd implantation dose increasing, while those of Al-12Si-2Mg/C(Si)-terminated SiC systems gradually increased, which can be mainly attributed to the variation of interfacial interactions of Al-12Si(-Mg)/SiC systems.

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#### 1. Introduction

Silicon carbide (SiC) is one of the most widely applied semiconductor materials in the high power, high frequency and high temperature semiconductor devices [1–3]. The wetting of metal/SiC system plays a crucial role in the fabrication of the SiC-reinforced metal-matrix composites, brazing of SiC ceramics and packaging the contact angle ( $\theta$ ), which is related to the bonding quality of the metal/ceramic interface. According to Young's equation:  $\cos\theta = (\sigma_{SV} - \sigma_{SL})/\sigma_{LV}$  (where  $\sigma_{ij}$  denotes the characteristic surface energies of the solid (S)-liquid (L)-vapor (V) system), two main technological approaches can be used to improve the wettability of metal/SiC system: (i) modifying the ceramic surface (such as ion implantation [4], electroless plating [7,8] sintering metallization [9], and plasma pulses techniques [10]) to increase the  $\sigma_{SV}$  of substrates; (ii) adding active elements (such as Ti [11], Cr [12], Si [13–15] and Mg [16–19]) to the metal for improving the interfacial interactions between molten metals and substrates to decrease the

of SiC semiconductor devices [4–6]. Generally, the wettability of a liquid metal on a solid ceramic surface is estimated by measuring

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 $\sigma_{SL}$  of the system. Especially, high dose and/or high energy of ion implantation into ceramic monocrystal substrate can cause high concentration of defects (lattice damage or disorder and vacancies), resulting in the increase of solid-vapor surface energy ( $\sigma_{SV}$ ) and introduce of implanted impurity atoms [20], which can affect the wettability of molten metals on the ceramic monocrystal substrate. For instance, our group investigated the influences of Mo ion implantation on the wetting kinetics of pure Ni and Ni-56Si alloy on SiC single crystal, indicating that the equilibrium or final contact angles decreased from 27° (for Ni-56Si/SiC) and 17° (for Ni/SiC) to <10°, respectively [4]. Moreover, it was reported that the addition of Mg can improve the wettability of Al/SiC system due to the reduction of  $\sigma_{IV}$ , promotion of the chemical interactions to reduce  $\sigma_{SL}$  and the stronger affinity for SiC [16–18]. However, the experimental results showed that the 7.6 wt% addition of Mg increased the equilibrium contact angle of Al/SiC system at 1173 K to a certain extent [18].

In addition, the surface polarity or orientation of SiC ceramic substrate may also play an important role in the wettability of metal/ceramic system. For instance, Shen [21] investigated the influence of SiC surface polarity on the wettability and reactivity in an Al/6H-SiC system at 973—1173 K, and found all the equilibrium or final contact angles of molten Al on C-terminated SiC were lower than those on Si-terminated SiC; however, the wettability of molten Al-12Si on the Si-terminated SiC was better than that on the C-terminated SiC at 1173 K.

In this work, the influences of SiC surface polarity and Pd ion implantation on the wettability of Al-12Si(-2 Mg)/SiC systems were investigated, and the interfacial behavior and microstructures were analyzed and discussed.

#### 2. Experimental procedure

The double-side polished C-terminated and Si-terminated 6H-SiC (0001) monocrystal wafers (Tankeblue Corp., Beijing, China) were used as ceramic substrates. The orientation of the SiC single crystals were  $\langle 0001 \rangle \pm 5^{\circ}$ , and were cut into 7 mm  $\times$  7 mm  $\times$  0.33 mm square pieces. The normal compositions of two Al alloys were Al-12Si and Al-12Si-2Mg (all in wt. %), with purities of 99.3% and 99.1%, respectively. Before the wetting tests, the two alloys were cleaned firstly in sodium hydroxide solution, while the SiC substrates were cleaned in dilute hydrochloric acid. And then both the metals and SiC substrates were ultrasonically cleaned in ethyl alcohol.

The ion implantation was carried out in an ion implantor (MEVVA-36, Beijing, China). Three implantation doses ( $5 \times 10^{15}$ ,  $5 \times 10^{16}$  and  $5 \times 10^{17}$  ions/cm<sup>2</sup>) of Pd ions were implanted into the SiC substrates at an energy of 20 keV at room temperature under a vacuum of  $\sim 6 \times 10^{-4}$  Pa. The Al-12Si and Al-12Si-2Mg alloys for the sessile drop tests were cut from the corresponding wires of Ø 2-3 mm and then ground into samples with dimensions of  $1 \text{ mm} \times 1 \text{ mm} \times 1 \text{ mm}$  for each wetting experiment. All the samples were placed into the furnace before heating. Subsequently, the furnace was heated to 1323 K at 5 K/min, held for 90-400 min in a vacuum of  $\sim 4 \times 10^{-4}$  Pa, and then furnace-cooled to room temperature. The wetting and spreading kinetics were evaluated by contact angle measurements with a precision of  $\pm 1.5^{\circ}$  using the sessile drop technique (OCA15LHT-SV, Dataphysics, Germany) in conjunction with the specially designed SCA image analysis software, here all the obtained contact angles were the average values between left and right ones. To ensure the molten sessile droplet can contact the SiC substrate directly, we selected the relatively high temperature of 1323 K to disrupt the dense oxide films (Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>) on the droplet and SiC substrate surfaces. The experimental procedure was described in detail in the previous work [9]. After the wetting tests, some couple samples were selected and immersed in saturated NaOH distilled-water solution to dissolve the solidified Al-12Si or Al-12Si-2Mg drop in order to expose the interface beneath the drop after they were mechanically removed the most part. The interfacial microstructures were observed and analyzed by scanning electron microscope (SEM) coupled with energy dispersive spectroscope (EDS).

#### 3. Results and discussion

#### 3.1. Effect of SiC surface polarity

Fig.1a shows the variations of the contact angle  $(\theta)$ , droplet diameter (D) and height (H) with time for the Al-12Si alloy on the as received C-terminated 6H-SiC substrate at 1323 K, respectively. As indicated, according to the variations of contact angle and droplet geometry, the wetting curve can be characterized by three stages: (i) the early pinning stage; (ii) the spreading stage; (iii) the final equilibrium stage.

For the Al-12Si/C-terminated SiC system, in the first stage all the  $\theta$  and the droplet sizes remained almost constant, and the relatively large initial contact angle (~160°) was attributed to the oxide films (Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>) on the Al-12Si alloy and SiC substrate surfaces [22]. Although all the samples were cleaned carefully before the wetting tests, it is still inevitable that the Al-12Si alloy surface was covered by alumina (Al<sub>2</sub>O<sub>3</sub>) during heating, which can prevent the contact between the molten drop and SiC substrate. Then, the triple line started to advance sharply with increasing the temperature and the second stage (ii) began, in which the  $\theta$  and H decreased while the D increased. In this stage, the spreading kinetics was mainly controlled by interfacial interactions, and the following chemical reactions can occur:

$$4Al_{(l)} + 3SiO_{2 (film)} = 2Al_{2}O_{3} + 3[Si]$$
 (1)

$$4Al_{(1)} + 3SiC_{(s)} = Al_4C_{3(s)} + 3[Si]$$
 (2)

According to Lee's experimental results and calculation, the addition of over 13.2 wt % Si into Al could entirely prohibit the formation of Al<sub>4</sub>C<sub>3</sub> in the Al/SiC system at 1323 K [23]. In this case, the Si concentration was 12 wt % which is a little less than the equilibrium Si content of Al-Si/SiC system at 1323 K. So, the spreading process in this stage was controlled by the reaction (1), namely the surface deoxidization of the substrate, and reaction (2). Thereafter, the  $\theta$ , D and H kept invariable, meaning that the system reached the final equilibrium stage with contact angle of ~31°.

The wetting of Al-12Si-2Mg/SiC system was similar with that of Al-12Si/SiC system. In the early stage, all the parameters  $(\theta, D, H)$  kept instant due to the presence of oxide films on the drop and substrate. During the stage (ii), following chemical reactions besides the reactions (1) and (2) can take place:

$$2 Mg_{(1)} + SiO_{2 (film)} = 2 MgO_{(s)} + Si$$
 (3)

$$2 Mg_{(1)} + Si_{(s)} = Mg_2Si_{(s)}$$
 (4)

Thermodynamically, the changes in the standard Gibbs free energy,  $\Delta G^0$ , for the chemical reactions (3) and (4) at 1323 K are -239.1 and -59.8 kJ mol $^{-1}$  [24], respectively, indicating that the two reactions are favorable at 1323 K. As the thin  $Al_2O_3$  oxide film was disrupted, the molten drop can contact with SiC substrate directly, the reactions (1–4) between the Al-12Si-2Mg and SiC would occur, resulting to an appreciable spreading. Similarly, the wetting of Al-12Si-2Mg/SiC system arrived at the equilibrium when the  $\theta$ , D and H did not change any more.

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