

## Wetting of pure aluminium on graphite, SiC and Al<sub>2</sub>O<sub>3</sub> in aluminium filtration

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**Abstract:** The wettability of pure aluminium on filter materials and on inclusions is believed to be an important factor affecting the filtration of aluminium. The contact angles of molten aluminium on alumina, SiC and graphite were measured under  $10^{-8}$  bar high vacuum in the temperature range of 1000–1300 °C. To describe the wetting behaviour of the Al on ceramic at lower temperatures used in filtration and casting aluminium, a semi-empirical calculation was employed. The calculated contact angles at 700 °C were around 97° for alumina, 92° for vitreous graphite, 126° for single- and poly-crystal graphite, and 79° for single crystal SiC, respectively. This indicates that aluminium does not wet alumina or graphite (or Al<sub>4</sub>C<sub>3</sub>) around the casting temperature, but wets SiC at this temperature. Thus a priming height is required for aluminium to infiltrate an alumina filter. Increasing temperature can also improve the wettability of Al on ceramic.

**Key words:** wettability; contact angle; aluminium; alumina; SiC; graphite

### 1 Introduction

Understanding the wetting behaviour of aluminium on ceramics is crucial for the improvement of the aluminium filtration process. Alumina is the most common filter material and SiC also can be a filter choice. Alumina and Al<sub>4</sub>C<sub>3</sub> are the typical inclusions in molten aluminium. The wettability of molten aluminium on these materials is the main subject of the present study.

The contact angles between alumina and molten aluminium are used to characterize the wetting behaviour. The wettability between aluminium and alumina should throw a light on how the metal and alumina inclusions or metal and alumina filter material interact. In the past decades, great effort has been paid to determining the wettability between molten aluminium and solid alumina [1–11] at temperatures of interest (as shown in Fig. 1). However, there is a considerable spread in the experimental results. Therefore, the contact angle under high vacuum conditions is measured, where the oxide film on aluminium can be eliminated.

All the measurements shown in Fig. 1 were carried

out under vacuum ( $10^{-9}$ – $10^{-7}$  bar), except those by JOHN and HAUSNER [1] who measured the contact angle under argon atmosphere under  $10^{-49}$  bar oxygen partial pressure. NAIDICH et al [4] reported their experimental results but did not mention the vacuum conditions.

Even though the contact angles tend to decrease with the increase of temperature, the results are rather scattered. For example, contact angles at 700 °C measured by different authors were in the range of 88°–167°. The scatter may be due to variations in the specimens (different purity, pre-treatment, crystallization, surface orientation, etc) used in the measurements and dissimilar experimental conditions (vacuum, oxygen potential and material in the furnace tube). The high vacuum work tends to give low contact angles, as indicated by the solid dots below  $10^{-9}$  bar vacuum shown in Fig. 1.

To our knowledge, the aluminium sessile drop is always covered by an oxide layer when pure argon under  $10^{-22}$  to  $10^{-21}$  bar oxygen partial pressure is employed. This leads to the anomalous high contact angle. For instance, a contact angle of 150° was measured at 1000 °C for both Al–graphite and Al–Al<sub>2</sub>O<sub>3</sub> system in

our early experimental work [12].

Aluminium is readily oxidized even if the oxygen partial pressure is as low as  $10^{-49}$  bar at 700 °C, as expressed by reaction (1) and shown in Fig. 2 [13]. Such a low oxygen partial pressure is difficult to achieve experimentally. Nevertheless, the oxide layer on the surface of a molten aluminium drop can be removed, if the outgoing flow of gaseous  $\text{Al}_2\text{O}$ , according to reaction (2), is greater than the incoming flow of oxygen. The equilibrium partial pressure of  $\text{Al}_2\text{O}$  (Fig. 2), according to reaction (2), is  $4.3 \times 10^{-5}$  bar at 1000 °C. Since the total pressure in the furnace is held under  $10^{-8}$  bar, the oxide skin on the aluminium drop is removed. This allows the

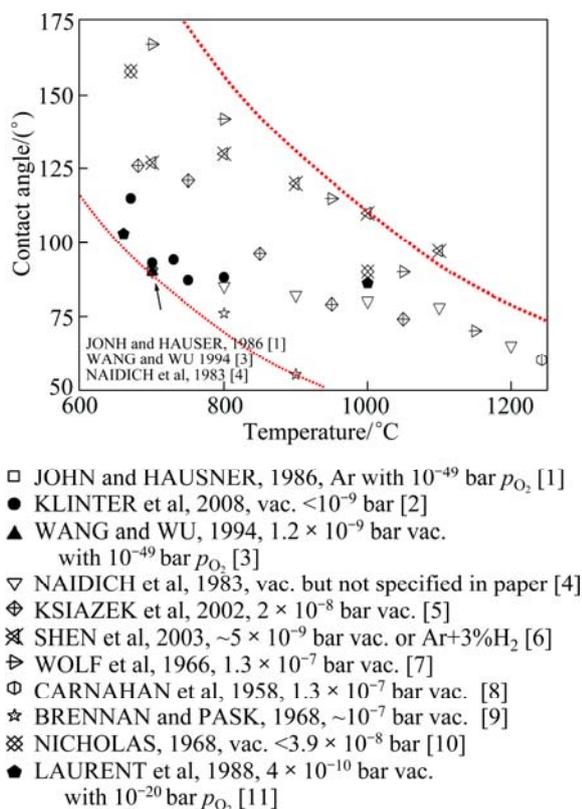


Fig. 1 Contact angles of Al on  $\text{Al}_2\text{O}_3$  at various temperatures [1–11]

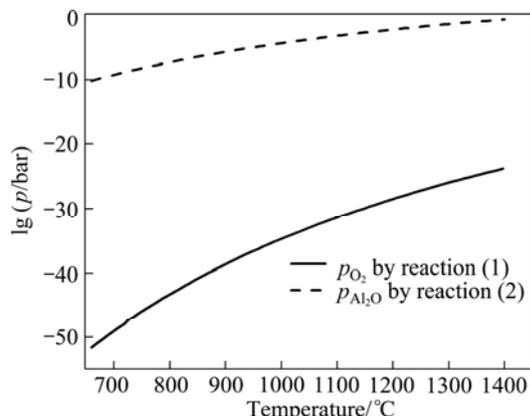


Fig. 2 Equilibrium partial pressure of gas species in reactions (1) and (2)

contact angles between molten aluminium and solid alumina can be measured.



Most of the published results obtained under low total pressure,  $10^{-9}$  bar, give low contact angles. It may be assumed that the oxide on the aluminium has been removed according to reaction (2) and the contact angle for the Al– $\text{Al}_2\text{O}_3$  system is measured.

The wettabilities of aluminium on single crystal SiC [14], reaction bonded SiC [15] and sintered SiC [15,16] were reported in the literature and summarized in Fig. 3. The difference in wettability depends mainly on the preparation process and the sintering aids for SiC. Aluminium wets the reaction bonded SiC better than the single crystal SiC or sintered SiC. For example, the measured contact angles for reaction bonded SiC, single crystal SiC and sintered SiC are 41°, 60° and 107° at 830 °C, respectively.

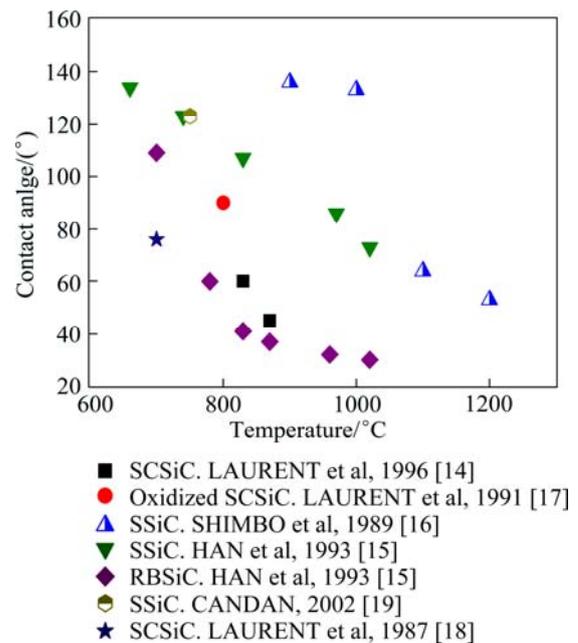
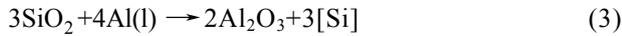


Fig. 3 Contact angles of Al on SiC at different temperatures [14–19] (SSiC–sintered SiC; RBSiC–reaction bonded SiC; SCSiC–single crystal SiC)

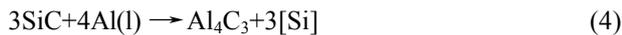
In aluminium filtration, filters are primed to allow metal to flow through the filter without freezing. The pre-heating temperature may reach the casting temperature and the SiC filter surface would be oxidized. This oxidation may affect the wetting behaviour. The oxidation of SiC is very slow at room temperature [20], but SiC will react with air to form a silica-rich surface layer at temperatures above 700 °C. SiC oxidation is controlled by the diffusion of oxygen molecules/ions through the thin oxide film [21]. The oxidation behaviour

of SiC is also influenced by factors such as moisture, SiC particle size and metal impurities [21]. LAURENT et al [17] found that silica acts as an oxygen donor to extend the life time of the alumina layer on an aluminium drop as:



This reaction does not improve the wetting of aluminium on SiC. See the circle marker ( $90^\circ$ ) at  $800^\circ\text{C}$  in Fig. 3, which is greater than  $76^\circ$  on the non-oxidized SiC by the same author at  $700^\circ\text{C}$ . The silica layer is changed into alumina and the equilibrium contact angle is the same as aluminium on alumina. With prolonging time in high vacuum, a thin initial layer of silica can be removed following reactions (3) and (2), successively. Aluminium is then in direct contact with SiC. The role of the silica here is to delay the wetting of aluminium on SiC [17].

When the liquid composition reaches the peritectic point in the Al–Si–C system at  $650^\circ\text{C}$ , the following ternary quasi-peritectic reaction [18] occurs isothermally as:



Addition of Si to aluminium prevents the formation of  $\text{Al}_4\text{C}_3$  and does not affect the wettability [18,22]. Also free Si in reaction bonded SiC prevents the formation of  $\text{Al}_4\text{C}_3$  [23] and effectively promotes wetting by aluminium in the temperature range of  $700\text{--}1040^\circ\text{C}$  [15]. This may be due to the additional Si–Al on the Al–SiC interface and the aluminium penetration into the SiC along the inter-granular free silicon resulting from the reaction bonding process in the extensive reaction zone [24].

Carbon filters were first industrialized as petrol coke filter-DUFI during 1970–1985, known as removing hydrogen, alkaline metals and non-metallic inclusions in plants operated by the ALUSUISEE Group and elsewhere [25]. However,  $\text{Al}_4\text{C}_3$  may form in a reaction between aluminium and graphite. The  $\text{Al}_4\text{C}_3$  crystals are brittle and highly sensitive to moisture and promote accelerated fatigue crack growth rates due to their hydrophilic nature [26].  $\text{Al}_4\text{C}_3$  inclusions in aluminium are a problem. The wetting behaviour of the Al–graphite (or Al– $\text{Al}_4\text{C}_3$ ) system will be investigated from the view

point of inclusion–metal wettability in filtration.

During the last decades, several studies have been devoted to the wetting behaviour of the Al–graphite system. As a reactive wetting system, it is agreed that: 1) the final or steady contact angle is equal or close to the equilibrium contact angle of the liquid on the reaction product,  $\text{Al}_4\text{C}_3$  [27]. The wetting behaviour of the reaction product is governed by the formation of adsorption layers at the interface, rather than by the subsequent nucleation and growth of the reaction product [27]. 2) The wettability would not be improved by the chemical reaction itself. An exchange of atoms is involved in a chemical reaction [28]. In the Al–graphite system, the final contact angle on the reaction product  $\text{Al}_4\text{C}_3$  is much lower than the initial contact angle on the original substrate graphite.

Aluminium oxide covers the molten aluminium in the launder in the casting house. When aluminium enters a filter, the oxide layers on the aluminium are either broken up into particles (inclusions) or they could cover parts of the filter. In priming a filter metal, the oxide films covering the filters must be penetrated and broken. Once the metal has entered the filter, oxidation is not a problem. Initially, molten aluminium is in contact with the filter covered with alumina. The wetting between aluminium, filter ceramics and inclusions was studied in the laboratory under high vacuum conditions.

In the present study, the contact angle of molten aluminium on ceramics in the temperature range of  $1000\text{--}1300^\circ\text{C}$  was measured using the sessile drop technique. The contact angle of Al–ceramic was extrapolated down to  $700^\circ\text{C}$  using a semi-empirical equation developed based on classic wettability theory.

## 2 Experimental

The experimental apparatus is schematically shown in Fig. 4. The apparatus essentially consists of a horizontal graphite heater surrounded by graphite radiation shields, located in a water-cooled vacuum chamber. The chamber was fitted with windows to allow a digital video camera (Sony XCD–SX910CR) to record the shape of the droplet. The contact angles and linear dimensions of the images were measured directly from

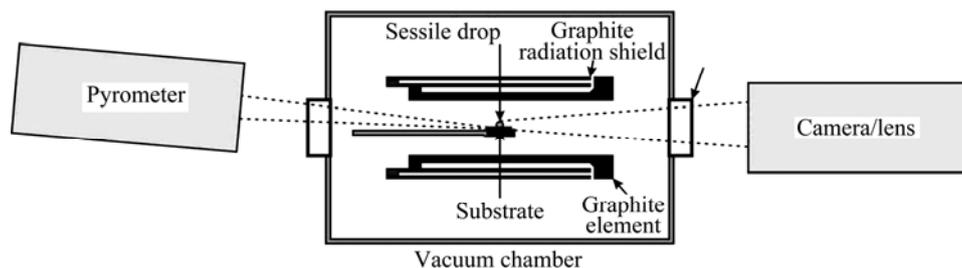


Fig. 4 Schematic diagram of sessile furnace

the image of the drop using Video Drop Shape Analysis software [12]. The symmetry of the drop was assumed. After the experiments, no asymmetry was observed.

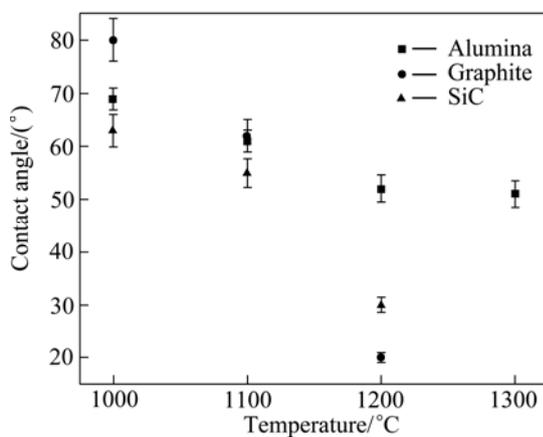
The experiments were carried out on wetting of 99.999% pure aluminium on 99.7% alumina, graphite (ISO-88) and 98.9% single crystal SiC (Washington Mills, Norway). Aluminium rod with diameter of 2 mm was cut into small pieces of around 2 mm in length, then polished by No. 500 sandpaper and cleaned with ethanol in order to prevent further oxidation. The average roughness of the substrates was 393.13 nm for alumina, 51.25 nm for SiC and 179.76 nm for graphite, respectively. When the wetting furnace attained the high vacuum of  $10^{-8}$  bar, the sample was quickly heated to 950 °C in about 80 s, then heated to 1000, 1100, 1200 and 1300 °C at a heating rate of 50 °C/min. In all the experiments, the contact angle and linear dimensions of the droplet were recorded simultaneously during the isothermal period at 1000, 1100, 1200 and 1300 °C.

### 3 Results and discussion

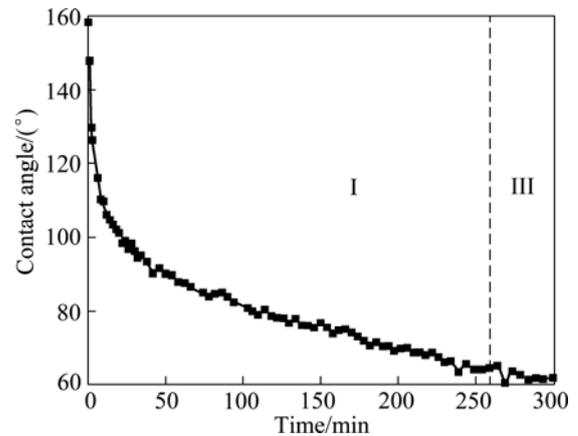
#### 3.1 Results

Contact angles measured on ceramics in the temperature range of 1000–1300 °C are summarized in Fig. 5. It can be seen that the wettability improves with temperature. Great effort was made to obtain contact angles at even higher temperatures, such as, 1400 °C under vacuum condition. Unfortunately, rapid evaporation affects the reliable results. As examples, the contact angles for Al–ceramics at 1100 °C are shown in Figs. 6–10.

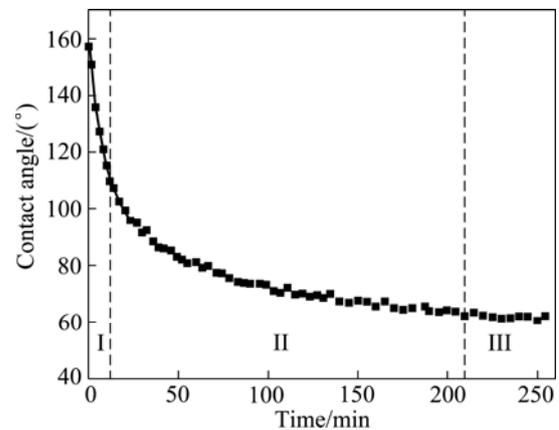
Figure 6 shows the time dependency of wetting properties for aluminium on solid alumina under  $10^{-8}$  bar vacuum at 1100 °C. The contact angles decrease from 158° to approximately 62° in 259 min. This period can be marked as stage I. Removal of the oxide layer and



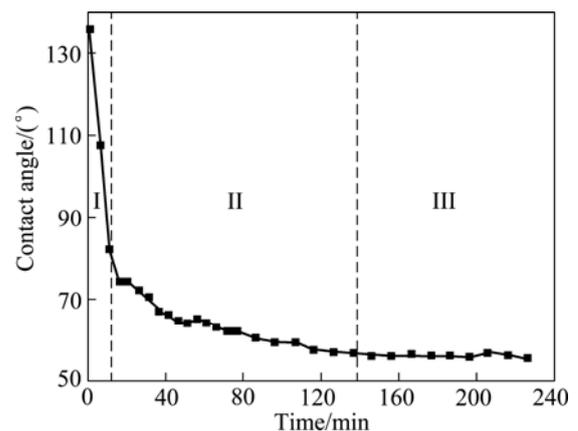
**Fig. 5** Contact angle vs time for Al on ceramics at various temperatures



**Fig. 6** Contact angle vs time for Al on  $\text{Al}_2\text{O}_3$  at 1100 °C



**Fig. 7** Contact angles vs time for Al on graphite at 1100 °C



**Fig. 8** Contact angles vs time for Al on SiC at 1100 °C

evaporation of molten aluminium explain the reduction of the contact angle. In the final stage, a relatively stable contact angle of approximately 62° is obtained. A decreasing sessile drop volume in stage I indicates that the reduction of oxide layer and evaporation of aluminium take place simultaneously. In the final stage, the base diameter becomes nearly stable and the equilibrium contact angle is obtained.

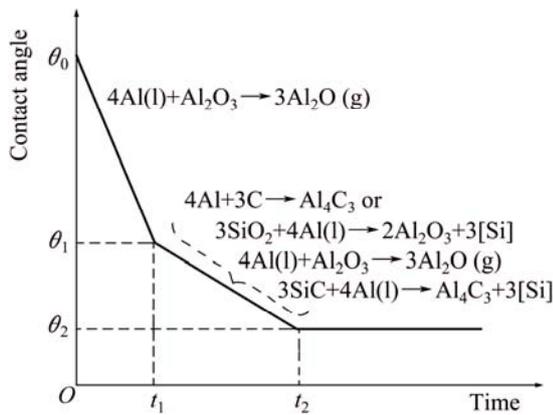


Fig. 9 Time dependent graph of related reactions

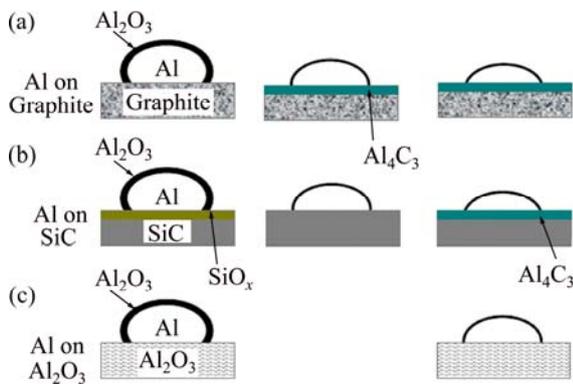


Fig. 10 Wettability of Al on ceramic: (a) Graphite; (b) SiC; (c) Al<sub>2</sub>O<sub>3</sub>

Figure 7 shows the time dependency of wetting properties for liquid aluminium on graphite at 1100 °C. The three kinetic stages can be distinguished as the first stage where the contact angle decreases rapidly from the initial contact angle 157° to 107° in 14 min, the second stage where the contact angle continues to decrease but at a slower rate, and the third stage corresponding to a nearly constant angle 62° at time longer than 209 min.

Figure 8 shows the time-dependent variations of wetting properties for liquid aluminium on SiC at 1100 °C. The three kinetic stages can be distinguished as the first stage where the contact angle decreases rapidly from 136° to 82° in 11 min, the second stage where the contact angle continues to decrease to a relatively low value in 136 min, but at a slower rate, and the third stage where the contact angle stabilized at around 55°.

### 3.2 Discussion

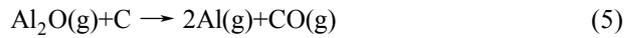
The three successive stages of wetting kinetics in Figs. 6–8 are discussed successively.

#### 3.2.1 Stage I—De-oxidation of oxide layer

The sharp decrease of the contact angles on both Al–SiC and Al–graphite is similar to the contact angle curve observed for Al–Al<sub>2</sub>O<sub>3</sub> at the same stage in the same wetting furnace. This reduction is due to the

de-oxidation of the oxide layer according to reaction (2). This is effective when the outgoing flow of oxygen in Al<sub>2</sub>O (g) is higher than the incoming flow of oxygen. The  $p_{total}$  of  $10^{-8}$  bar in the furnace means that  $p_{Al_2O}$  is lower than the equilibrium  $p_{Al_2O}$  of  $3.7 \times 10^{-8}$  bar at 1000 °C [13]. Thus, the wetting behaviour of the first stage is controlled by de-oxidation of the oxide layer on the aluminium drop.

The time needed for de-oxidation in stage I for Al–Al<sub>2</sub>O<sub>3</sub> system is longer than that of the other two systems. There are several reasons. In the Al–Al<sub>2</sub>O<sub>3</sub> system, the vacuum removing oxide from the aluminium drop according to reaction (2) also has to deal with the alumina substrate. In the other two systems, carbon helps to remove the oxide layer by reaction



#### 3.2.2 Stage II—De-oxidation of silica in Al–SiC system and Al<sub>4</sub>C<sub>3</sub> formation in Al–graphite system

There are three reasonable explanations concerning the second stage in Al–SiC system: 1) dissolution of SiC into aluminium according to reaction (6), 2) coverage of the interface by Al<sub>4</sub>C<sub>3</sub> according to reaction (4), and 3) de-oxidation of silica on the interface according to reactions (3) and (2).

The maximum solubility of carbon in liquid aluminium, on the order of  $30 \times 10^{-5}$  at temperatures around 1000 °C [29] is too low to support the dissolution mechanism. The kinetics of reaction (4) is slow and leads to the limited formation of discrete particles of Al<sub>4</sub>C<sub>3</sub> at the interface. Thus the spreading of de-oxidized aluminium is controlled by the de-oxidation of SiC with the formation of limited amounts of discrete particles of Al<sub>4</sub>C<sub>3</sub>. This is supported by the delayed equilibrium contact angle obtained in the Al-oxidized SiC system [17] at 660–900 °C.



In the second stage in Al–graphite system, the spreading velocity is lower than that at the previous stage and the base diameter is a linear function with respect to time. This second stage does not exist in the non-reactive Al–Al<sub>2</sub>O<sub>3</sub> system. The interfacial reaction (7) has Gibbs energy of –136 to –102 kJ/mol [30] at temperature from 660 to 1300 °C. An Al<sub>4</sub>C<sub>3</sub> layer at the Al–graphite interface observed by SEM also supports the assumption that the second stage in the Al–graphite system is controlled by the formation of Al<sub>4</sub>C<sub>3</sub>.

Note that for the Al–Al<sub>2</sub>O<sub>3</sub> system, there is not such a stage.

#### 3.2.3 Stage III—Stable contact angle

The Al–Al<sub>4</sub>C<sub>3</sub> system is formed at the end of the

second stage in both the Al–graphite and Al–SiC systems. A relatively stable contact angle is detected in the third stage for all these 3 systems. Good repeatability of wetting properties is observed at the same temperatures.

### 3.2.4 Schematics of stages I to III

To summarize the discussion, the reactions and schematic diagram of the wettability for the Al–ceramic systems is shown in Figs. 9 and 10. The contact angle initially changes with time and finally approaches an equilibrium value. Both Al–SiC and Al–graphite systems produce  $Al_4C_3$  at the end and the interface reaction product promotes wetting. However, silicon in aluminium could prevent the  $Al_4C_3$  formation in the Al–SiC system.

### 3.3 Calculation of contact angle

The present measurements were carried out at relatively high temperatures in order to minimize the effect of the aluminium oxide layer. Taking the Al– $Al_2O_3$  system as an example, the measured high temperature contact angle down to 700 °C is extrapolated.

The equilibrium value of the contact angle  $\theta$  which is used to define the wetting behaviour of the liquid, obeys the classical equation of Young (see Eq. (8)). The effect of the curvature of the aluminium droplet is not taken into account [31,32].

Most of the reported surface tension measurements pertain to “oxygen saturated” samples [33]. According to MILLS and SU [33], the surface tension of oxygen saturated [33] molten aluminium is given by Eq.(9).

$$\cos \theta = (\sigma_{SV} - \sigma_{SL}) / \sigma_{LV} \quad (8)$$

$$\sigma_{LV} = 1043 - 0.18T \text{ (mN/m)} \quad (9)$$

where  $\sigma$  is the surface tension; the subscripts S, V and L refer to the solid, vapor and liquid, respectively;  $T$  is thermodynamic temperature.

EUSTATHOPOULOS et al [34] reported the experimental surface energies of alumina using the multiphase equilibrium technique. The extrapolation of the experimental data to low temperatures gives a value of  $(1400 \pm 500)$  mN/m, which roughly agrees with the first principle simulation results [35], as shown in Fig. 11.

The work of adhesion,  $E_A$ , can now be obtained from Eq.(10). GIRIFALCO and GOOD [36] proposed that the work of adhesion was proportional to the geometric average of the surface energies of two media, see Eq.(11).

$$E_A = \sigma_{LV} + \sigma_{SV} - \sigma_{SL} \quad (10)$$

$$E_A = 2\phi \sqrt{\sigma_{LV} \sigma_{SV}} \quad (11)$$

where  $\phi$  is a function of the molar volumes of the liquid and the solid. In the present work, the deviation between

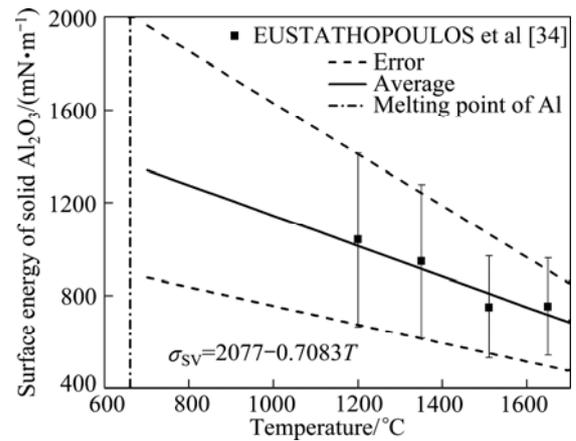


Fig. 11 Surface energy of solid  $Al_2O_3$  at various temperatures

the calculated and measured interfacial energies is minimized regarding the coefficient  $\phi$  as being temperature- dependent. Figure 12 shows the fitted line for the coefficient  $\phi$ . The proportionality factor increases monotonically with temperature. The experimental results at low temperatures reported by JOHN and HAUSNER [1], KLINTER et al [2] and WANG and WU [3] have also been included. They all achieved a total pressure lower than  $10^{-9}$  bar, except those by JOHN and HAUSNER [1]. It is found that

$$2\phi = 1.55 - 14.38 \exp(-0.0029T) \quad (12)$$

The equation is applicable for the temperature range of 700–1500 °C. The interfacial energy of aluminium and alumina is obtained when  $\sigma_{LV}$ ,  $\sigma_{SV}$  and Eq.(12) are inserted into Eqs. (10) and (11). At the melting point of aluminium,  $\sigma_{SL} = 1.63 \text{ J/m}^2$  is obtained. This value is in good agreement with the value of  $1.66 \text{ J/m}^2$  reported by NIKOLOPOULOS et al [37].

From the above equations, the contact angles of the aluminium and alumina at various temperatures under vacuum can be calculated using Eq. (8). Figure 13 shows

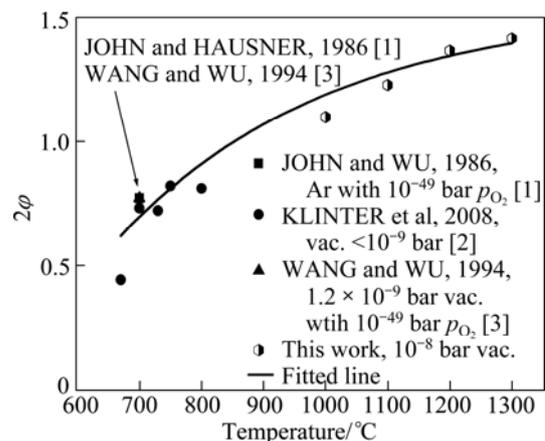
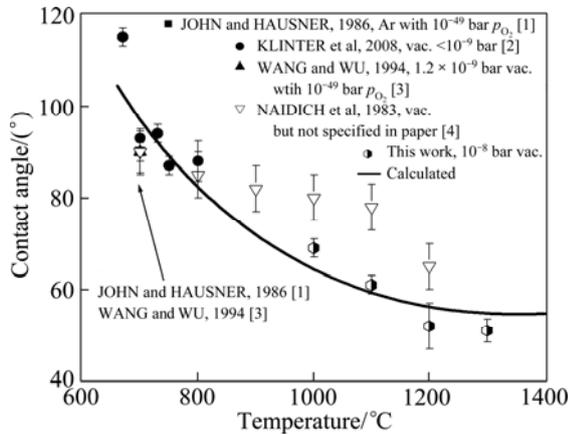


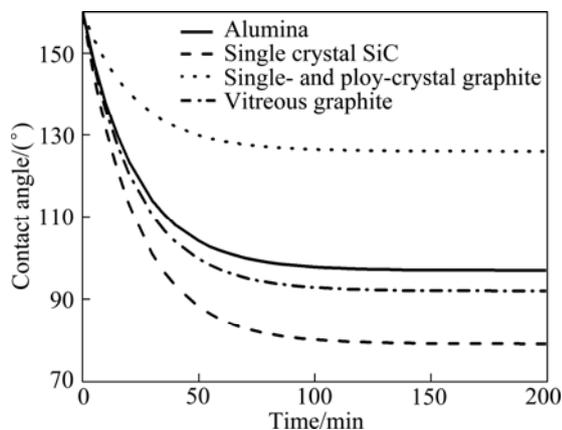
Fig. 12 Coefficient  $\phi$  fitted to literature data and this work

the calculated contact angle with the measured values at various temperatures. The contact angle between aluminium and alumina at 700 °C is around 97°, which indicates that alumina is not wetted by molten aluminium at the casting temperature.



**Fig. 13** Calculated and measured contact angles vs temperature for Al on Al<sub>2</sub>O<sub>3</sub>

Similar calculations have been performed for the Al–SiC and Al–graphite systems. The contact angles at 700 °C are 79° for single crystal SiC, 92° for vitreous graphite, and 126° for single- and poly-crystal graphite, respectively. The time-dependent wettability of aluminium on various materials is compared in Fig. 14. The contact angle decreases with time and SiC has the best wetting with aluminium among these systems. Note that the same de-oxidation time is employed here for comparison. However, the Al–SiC and Al–graphite systems should have faster de-oxidation of the oxide layer in stage I.



**Fig. 14** Contact angle of Al on various materials at 700 °C

It is seen that the wetting of the Al–ceramic system improves with increasing temperature. It may take considerable time to improve the wetting between metal

and filter. To initiate the filtration of aluminium, it may be necessary to use a priming height up to 400 mm [38,39] or to heat the filter and the filter bowl to the temperature above the melting point of aluminium. In our filtration pilot trials, the metal height above the filter is around 200 mm. Once the metal has infiltrated the filter, the metal height can be reduced.

The inclusions in aluminium entering the filter may have been in the metal for a longer time giving better wetting. This is a disadvantage for the removal of the filter.

The refining of aluminium by filtration is mainly carried out in 1 bar atmosphere when the liquid aluminium is definitely covered with oxide films. The formation of oxide films is certain with  $2 \times 10^{-1}$  bar O<sub>2</sub> (1 bar atmosphere). Before filtration, priming (preheating of filter or filter bowl to avoid blockage) is performed. When the liquid aluminium flows into the filter, aluminium is oxidized by the air in the filter box and oxide film is captured by the filter. Thus, liquid aluminium contacts the interface covered with oxide film, only part of the ceramics. NI et al [40] suggests that a layer of oxide film exists between the melt and filter framework in the ceramic foam filter (CFF). When the first stream of melt comes into CFF, oxidation becomes more serious due to the very large inner surface area of CFF. Thus, the wettability of aluminium on oxide films and substrate is important in filtration. At 700 °C, the initial contact angle value is approximately 150° and roughly independent of the nature of the substrates [12,22].

## 4 Conclusions

1) The wetting behaviour between molten aluminium and ceramics in the temperature range from 1000 to 1300 °C is investigated. The contact angles decrease with increasing temperature. The contact angles of Al–Al<sub>2</sub>O<sub>3</sub>, Al–SiC and Al–graphite at 700 °C are calculated to be 97°, 79°, 92° (for vitreous graphite) and 126° (single- and poly-crystal graphite), respectively. This is in good agreement with experimental values in the literature.

2) Improved wetting of aluminium on ceramics with temperature is an advantage in getting molten metal to infiltrate alumina. In priming filters, it is necessary to have a metal height above the filter or to increase the temperature. However, filtration may proceed at lower temperature once the metal has entered the filter.

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## 纯铝与石墨、SiC 和 Al<sub>2</sub>O<sub>3</sub> 的润湿性

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**摘 要:** 纯铝与过滤材料和夹杂物之间的润湿性是影响铝的过滤性能的重要因素。在  $10^{-8}$  bar 真空条件下于 1000~1300 °C, 研究液态铝与氧化铝、SiC 和石墨之间的润湿角。为了描述铝的过滤和浇铸等在低温条件下铝与陶瓷的润湿行为, 使用半经验模型进行研究。计算出的 700 °C 的纯铝与氧化铝的润湿角为 97°, 与玻璃体石墨的为 92°, 与单晶和多晶石墨的为 126°, 与单晶的为 SiC 79°。结果表明: 在浇铸温度下铝不能润湿氧化铝和石墨(或者 Al<sub>4</sub>C<sub>3</sub>), 但可以润湿 SiC。因此, 为了让铝能够浸润过滤材料, 在铝过滤时需要施加一个压力差作为推动力。提高温度也可以改善铝与陶瓷间的润湿性。

**关键词:** 润湿性; 润湿角; 铝; 氧化铝; SiC; 石墨

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