



# Article **The Effect of Temperature and Sputtered Particles on the Wettability of Al/Al<sub>2</sub>O<sub>3</sub>**

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**Abstract**: Two kinds of  $Al_2O_3$  ceramic samples with and without Al film deposited were designed respectively. The influences of temperature and high kinetic energy sputtering particles on the wettability and interface strength of  $Al/Al_2O_3$  were studied by comparing the wetting behavior of molten aluminum on two samples. The results show that molten aluminum does not wet the  $Al_2O_3$  sample without Al film deposited at 700 °C, the contact angle is 165°, and the interfacial shear strength is 28 MPa. With the increase of temperature, the contact angle decreases continuously, and the interface shear strength gradually increases. The fracture of the brazed joint is transferred from the interface to the brazing seam. In comparison, the sample deposited with Al film is wetted by molten aluminum at 700 °C, and the contact angle is only 12°. The interface shear strength is about 120 MPa and is less affected by temperature. The shear fracture of the joint occurs in the brazed seam of Al metal. Therefore, the high energy generated by either the temperature increase or the particle sputtering enable the Al atoms to overcome the energy barrier to form Al–O bonds with the O atoms on the  $Al_2O_3$  ceramic surface, thereby improving the wettability of  $Al/Al_2O_3$ .

Keywords: wettability; interface strength; sputtered film; Al<sub>2</sub>O<sub>3</sub> ceramic; aluminum

# 1. Introduction

The wettability of the metal melt to the ceramic plays an important role in the preparation of composite materials and ceramic connection. As a typical non-reactive wetting system, there have been many research reports on the wettability of  $Al/Al_2O_3$ . However, due to the interference of the oxide film on the Al liquid surface, which is extremely difficult to remove, the wetting behavior is still insufficient. For example, there is a lot of controversy in the report on the change of wetting angle with temperature [1–9]. Some studies [5–7] believe that the freshly melted Al liquid cannot directly wet  $Al_2O_3$ , and the wetting angle is  $110^{\circ}-150^{\circ}$ . As the temperature increases, the wetting angle gradually decreases, and reduces to below 90° to achieve wetting after 850 °C. There are also reports [8,9] that this wetting temperature needs to be as high as  $1000-1050^{\circ}$ C, or even  $1200^{\circ}$ C.

Although the above studies are controversial on the wetting transition temperature of  $Al/Al_2O_3$ , most of them believe that the contact angle of Al to  $Al_2O_3$  ceramics will gradually decrease with the increase of the Al liquid temperature. Some studies have shown that the wettability of the  $Al/Al_2O_3$  is related to the bonding state of the atoms at the interface. Zhang et al. [10] studied the interfacial structure and wetting behavior of Al and  $Al_2O_3$  ceramics using molecular dynamic simulation methods. The results showed that during the heating process, as the thermal kinetic energy of atoms in Al melt increases, Al atoms gradually form Al–O chemical bonds with O atoms on the ceramic surface. The  $Al/Al_2O_3$  interface also changed from the physical adsorption state to the chemical



**Citation:** Li, Y.; Shang, H.; Ma, B.; Guo, X.; Li, R.; Li, G. The Effect of Temperature and Sputtered Particles on the Wettability of Al/Al<sub>2</sub>O<sub>3</sub>. *Materials* **2021**, *14*, 2110. https:// doi.org/10.3390/ma14092110

Academic Editor: Rainer Hippler

Received: 22 March 2021 Accepted: 18 April 2021 Published: 22 April 2021

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**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). bonding state, thereby improving the wettability of Al to Al<sub>2</sub>O<sub>3</sub> ceramics. The research of Shen et al. supports this theory experimentally [11]. They studied the wettability of Al<sub>2</sub>O<sub>3</sub> ceramics R-type (0112), A-type (1120), C-type (0001) crystal planes and polycrystalline Al<sub>2</sub>O<sub>3</sub> ceramics (PC) by Al melt. The results show that the order of wettability with molten Al is  $R \approx A > PC > C$  according to the strength of the atomic bond formed with Al atoms.

However, limited by the reduction of the evaporation temperature of molten Al in a high vacuum environment, most studies on the wettability of Al/Al<sub>2</sub>O<sub>3</sub> systems are below 1300 °C, and the lowest wetting angles obtained are also different. Some have just reached the wet state of about 90° [12–14], some have a better wet state of about 75° [15–18], and some may even reach about 55° [19]. The effect of higher kinetic energy of Al atoms on the wettability of Al/Al<sub>2</sub>O<sub>3</sub> remains to be further studied. The kinetic energy of sputtered Al particles is as high as  $10^0$  eV [20], which is 1–2 orders of magnitude higher than that of thermally evaporated Al. Therefore, it is expected to study the effect of higher Al atomic kinetic energy on the wettability of Al/Al<sub>2</sub>O<sub>3</sub> using a sputtering method.

In order to reveal the effect of Al atom kinetic energy on the wettability of  $Al/Al_2O_3$ , two kinds of  $Al_2O_3$  ceramic samples with and without Al film deposited were designed in this paper, respectively. The wetting behaviors of Al on these two  $Al_2O_3$  are studied using a sealed chamber that can obtain extremely low oxygen and nitrogen partial pressure. The effect of temperature and high kinetic energy of sputtered Al particles on the wettability of  $Al/Al_2O_3$  is revealed.

### 2. Materials and Methods

## 2.1. Sample Design

In order to reveal the wetting behavior of Al on Al<sub>2</sub>O<sub>3</sub>, two kinds of Al<sub>2</sub>O<sub>3</sub> ceramic samples with and without Al film deposited on the surface were designed, respectively. The schematic diagram of the structure is shown in Figure 1. In Figure 1a, there is no film deposited on the surface of the Al<sub>2</sub>O<sub>3</sub> ceramic sample, and a pure Al film with 1  $\mu$ m thickness is deposited on the surface of the Al<sub>2</sub>O<sub>3</sub> ceramic in Figure 1b.



**Figure 1.** The schematic of two kinds of Al<sub>2</sub>O<sub>3</sub> ceramic samples. (**a**) without Al film deposited; (**b**) with Al film deposited.

# 2.2. Thin Film Deposition

The 1 mm thick polished high-purity  $Al_2O_3$  ceramic substrate is ultrasonically cleaned in alcohol and placed on the substrate holder in the vacuum chamber of the multi-target magnetron sputtering system (SPC-350, Anelva, Tokyo, Japan). When the background vacuum of the vacuum chamber reaches  $5.0 \times 10^{-4}$  Pa, the ceramic substrate is baked at 400 °C × 10 min to remove the gas and impurities adsorbed on the surface. After the ceramic substrate is cooled to room temperature, Ar gas with a purity of 99.999% is filled into the vacuum chamber and the pressure is maintained at 0.6 Pa. The Ø 76 mm Al target (purity 99.99%) is controlled by a DC cathode. During the film deposition, the power of the DC cathode is maintained at 400 V × 0.5 A, and the substrate is neither heated nor applied with a negative bias. By controlling the deposition time, 1 µm thickness Al film is deposited on the  $Al_2O_3$  ceramic substrate.

## 2.3. Wetting Experiment

In order to eliminate the influence of  $O_2$  and  $N_2$  on the wetting behavior of Al after melting, the experiment adopted a sealed chamber that can cut off the oxygen and nitrogen sources. The structure of the sealed chamber is shown in Figure 2. A flat-mouthed quartz cup is buckled upside down on a quartz plate, and an aluminum-based solder plate is placed between them. The top of the quartz cup is pressed by a stainless steel weight block. When heated in a vacuum stove, the Al-Cu solder melted first and thus made the sealed chamber airtight, isolated from the outside vacuum environment. The residual  $O_2$  and  $N_2$  in the sealed chamber (including desorbed from the inner surface) would be gradually consumed due to the oxidation of the Al-Cu solder and aluminum powder. As a result, a very high vacuum was achieved.



Figure 2. The schematic of the sealed chamber.

During the wetting experiment, the Al<sub>2</sub>O<sub>3</sub> ceramic sample with and without Al film deposited is placed in this sealed chamber. A cleaned pure aluminum block with a size of  $2 \times 2 \times 2$  mm<sup>3</sup> is placed on the sample. The sealed chamber is placed in a vacuum furnace and evacuated to  $10^{-1}$  Pa. It is heated to 700, 800, 900, 1000, and 1100 °C respectively for 30 min and then cooled with the furnace. By comparing the wetting behavior of molten aluminum on the two samples, the effect of the kinetic energy of Al atoms on the wetting of Al/Al<sub>2</sub>O<sub>3</sub> is revealed.

# 2.4. Interface Strength Test

In our previous research, a kind of aluminum foil solder-coated bi-layer film was proposed to achieve direct brazing of ceramic without interfacial reaction layers [21]. For this paper, a 2  $\mu$ m Al layer followed by a 0.1  $\mu$ m Cu layer was deposited on both side of a 100  $\mu$ m pure aluminum foil. When melted, the coated foil would form an Al alloy liquid with 0.1 at.% Cu. Due to the low Cu content, it can be regarded as a pure Al melt. Holding the coated Al foil between two Al<sub>2</sub>O<sub>3</sub> substrates, a stainless steel weight was placed on top of the sample to fix and exert pressure. This brazing sample is placed flat in a vacuum furnace. When the furnace vacuum reaches 10<sup>-4</sup> Pa, the Al<sub>2</sub>O<sub>3</sub> ceramic samples are brazed at 700–950 °C for 30 min and then cooled with the furnace. The two kinds of Al<sub>2</sub>O<sub>3</sub> ceramic samples are brazed respectively with the coated aluminum foil solder to obtain a brazed joint containing an Al/Al<sub>2</sub>O<sub>3</sub> interface. Through the measurement of the joint shear strength and the observation of the fracture mode, the interface strength of Al/Al<sub>2</sub>O<sub>3</sub> was studied.

The structure and composition of the brazed joint are analyzed by scanning electron microscope (SEM, Hitachis-3400n, Hitachi, Tokyo, Japan) and the attached X-ray energy dispersive spectroscopy (EDS). The shear strength of the brazed joint is tested by an electronic tensile machine, and the size of the shear surface of the test sample is  $3 \times 2 \text{ mm}^2$ , where it was fixed in a special mold, as shown in Figure 3. The obtained shear strength is the average of the effective values of more than 10 samples. The morphology of the shear fracture is observed by SEM and optical microscope (OM, VHX-1000, Keyence, Osaka, Japan), whereas the composition is analyzed by EDS.



Figure 3. The fixture schematic diagram for measuring shear strength of brazed joints.

#### 3. Results

#### 3.1. Wetting Behavior

Figure 4 compares the wetting behavior of molten aluminum on two samples. All the aluminum blocks were eventually clustered into a spherical shape or spread on the  $Al_2O_3$  ceramic substrate. This result shows that the sealed chamber designed in this paper successfully cut off the channels for O<sub>2</sub> and N<sub>2</sub> to enter, and obtained a vacuum environment with extremely low oxygen and nitrogen partial pressure, preventing further thickening of the oxide film on the surface of the molten aluminum, and breaking the constraints of the surface oxide film. Therefore, the molten aluminum can flow and change shape under the action of surface tension. Figure 4a–e shows the wetting behavior of molten aluminum on samples of undeposited Al film Al<sub>2</sub>O<sub>3</sub> at different temperatures. The molten aluminum cannot wet Al<sub>2</sub>O<sub>3</sub> ceramics at 700 °C. This contact angle gradually decreases with the increase in temperature, and the wettability of molten aluminum to  $Al_2O_3$  ceramics is improved. The contact angle decreases to a critical value of about 90° when the temperature increases to 1000 °C. With a further increase in the temperature value of 1100 °C, the wettability of molten aluminum to Al<sub>2</sub>O<sub>3</sub> ceramics changes from non-wetting to wetting. However, the molten aluminum exhibits completely different wetting behavior on  $Al_2O_3$  ceramics with Al film deposited. As shown in Figure 4f, the molten aluminum is almost completely spread on this sample at 700 °C, indicating very good wettability.



**Figure 4.** The wettability of molten aluminum on the two kinds of sample at different temperatures. Without Al film deposited sample: (a) 700 °C; (b) 800 °C; (c) 900 °C; (d) 1000 °C; (e) 1100 °C; with Al film deposited: (f) 700 °C.

Figure 5 shows the contact angle change of molten aluminum on the two kinds of samples. For  $Al_2O_3$  ceramic samples without Al film deposited, the contact angle gradually decreases with the increase in temperature, from 165° at 700 °C to 90.8° at 1000 °C. With a further increase in the temperature to 1100 °C, the contact angle decreases to 80.3°. The



contact angle of molten aluminum on  $Al_2O_3$  ceramics deposited with Al film is as low as  $12^\circ$ , at 700 °C.

Figure 5. The contact angle change of molten aluminum on the two kinds of sample.

## 3.2. Interface Strength

Brazing experiments show that two  $Al_2O_3$  ceramic samples can be successfully brazed by Al foil solder at various temperatures. Through SEM observation, it is found that the brazed joints of the two kinds of sample obtained at different brazing temperatures have similar structures. Figure 6a shows the joint SEM morphology of the  $Al_2O_3$  ceramic sample without an Al film, which is brazed at 700 °C. The Al brazing seam in this picture is dense and full, and there are no brazing defects such as incomplete penetration and pores. In addition, it can be seen from the area-scanning of Al, O, and Cu (Figure 6b–d) that the interface between the brazing seam and the  $Al_2O_3$  ceramic is clear, and no element enrichment is found. This result indicates that there is not any reaction transition layer at the interface.



**Figure 6.** The joint SEM images of the  $Al_2O_3$  ceramic sample without Al film brazed at 700 °C (**a**) and corresponding area-scan images of Al (**b**), O (**c**) and Cu (**d**) elements.

Figure 7 shows the relationship between the brazed joints shear strength of the two kinds of sample and temperature. For  $Al_2O_3$  ceramic samples without Al film deposited, the shear strength of the joint brazed at 700 °C is 28 MPa. The contact angle gradually decreases with the increase in temperature. The shear strength of the joint increases with the increase in brazing temperature, and the increase rate slows down after reaching 115 MPa at 900 °C. It is worth noting that the shear strength of the brazed joint of the  $Al_2O_3$  ceramic sample with the Al film deposited shows a significantly different trend. It has reached a high value of about 120 MPa at 700 °C, and does not change significantly with the increase in brazing temperature.



**Figure 7.** The relationship between the brazed joints shear strength of the two kinds of sample and temperature.

The optical microscope observation of the shear fracture of each brazed joint (Figure 8) found that the fracture morphology of the Al<sub>2</sub>O<sub>3</sub> ceramic sample without Al film deposited greatly changes with the increase in the brazing temperature. The fracture of joints brazed at 700  $^{\circ}$ C is basically on the surface of Al<sub>2</sub>O<sub>3</sub> ceramics, and there are only a few small and scattered light-colored Al regions (Figure 8a). This shows that the fracture of this brazed joint occurs at the interface between the brazing seam and the ceramic. When the brazing temperature rises to 750 °C, the area of the light-colored Al increases (Figure 8b), indicating that partial fracture has occurred at the Al metal in the brazing seam. The area of the light-colored Al region in the 800 °C joint fracture continues to increase, and the furrow-like morphology along the direction of the shear force formed after the ductile Al shear fracture is observed (Figure 8c). When the brazing temperature is increased to 900 °C, the fracture of the joint is entirely composed of the furrow-like morphology of Al metal (Figure 8e). This series of changes shows that the shear fracture of the joint gradually shifts from the interface to the brazing seam as the temperature increases. Combining the wettability results in Figure 4 and the shear strength of the joint in Figure 7, the increase in the shear strength with the brazing temperature comes from the improvement of the wettability of Al<sub>2</sub>O<sub>3</sub> ceramics by molten aluminum, which increases the interfacial strength of  $Al/Al_2O_3$ .



**Figure 8.** Optical microscope images of the joints fracture morphologies of the two kinds of sample brazed at different temperatures. Without Al film deposited sample: (**a**) 700 °C; (**b**) 750 °C; (**c**) 800 °C; (**d**) 850 °C; (**e**) 900 °C; with Al film deposited: (**f**) 700 °C.

Figure 8f shows the shear fracture morphology of the  $Al_2O_3$  joint with Al film at 700 °C. Different from the  $Al_2O_3$  joint without Al film, this fracture morphology is composed of furrow-like morphology and does not change significantly with the increase in temperature. This result is also consistent with the joint shear strength in Figure 7. The joint shear strength has reached a high value of about 120 MPa at 700 °C, and does not change significantly with the increase in brazing temperature. Further combining the wettability results in Figure 4, it can be concluded that the Al film directly sputtered and deposited on the  $Al_2O_3$  ceramic has the effect of improving the wettability and interface strength.

Figure 9 shows the area-scanning results of the Al, O, and Cu elements of the brazing fractures of the two kinds of sample. A large amount of O elements and very few Alrich regions are distributed in the fracture of the undeposited Al film samples at 700 °C (Figure 9a). This result indicates that the fracture mainly occurs at the interface between the brazing seam and the ceramic. As the brazing temperature increases, the area of the Al-rich area on the fracture gradually increases, and the corresponding O element decreases. When the temperature increased to 900 °C, the fracture was mainly covered by Al element (Figure 9c), indicating that the fracture mainly occurred in the Al brazing seam. For the sample deposited with the Al film, the fracture at 700 °C is basically covered by Al (Figure 9d), which indicates that the fracture of the joint at this temperature has occurred in the Al weld.



**Figure 9.** The area-scanning results of the Al, O, and Cu elements of the brazing fractures of the two kinds of sample. Without the Al film deposited sample: (**a**) 700 °C; (**b**) 800 °C; (**c**) 900 °C; with Al film deposited: (**d**) 700 °C.

# 4. Discussion

The study of undeposited Al film samples in this article shows the effect of temperature on the wettability of  $Al/Al_2O_3$ . With the increase in the system temperature, the contact angle of molten aluminum to  $Al_2O_3$  decreases continuously, the  $Al/Al_2O_3$  interface strength gradually increases, and the shear fracture of the joint shifts from the interface to the brazing seam. The improvement of the wetting state mainly comes from the decrease in the liquid/solid interfacial tension of the system, which is essentially the Al-O chemical bond gradually formed between the Al atom in the molten aluminum and the O atom on the surface of  $Al_2O_3$ . According to the molecular dynamics study of Zhang et al. [10] and the first-principles study of Sun et al. [22], there are two binding states between the molten Al atom and the O atom on the ceramic surface. When the temperature of the system is low, physical adsorption with "gap" is formed between them, and as the temperature increases, a chemical bond with an Al–O chemical bond is formed. The formation of the Al-O chemical bond significantly reduces the interfacial tension between molten aluminum and  $Al_2O_3$ , which makes molten aluminum wetting  $Al_2O_3$ . The increase in temperature provides kinetic energy for Al atoms to overcome the barrier that forms the Al–O bond. It should be noted that wetting is a progressive process, because the formation of Al–O bonds is not only related to the kinetic energy of Al atoms, but is also related to the existence state of  $Al_2O_3$  terminal atoms. Pilania's research [23] shows that the contact angle of molten Al on different crystal planes of Al<sub>2</sub>O<sub>3</sub> is different at the same temperature.

The study of deposited Al film samples shows that the sputtering deposition of Al film can improve the wettability of  $Al/Al_2O_3$ . During magnetron sputtering, the kinetic

energy of gas phase particles (atoms or ions) sputtered from the cathode target surface is as high as  $10^0$  eV [20]. This energy is not only much higher than the thermal kinetic energy of molten aluminum atoms, but even an order of magnitude higher than the energy of Al atoms when they are thermally evaporated above 1000 °C. The impact of these high-energy sputtered Al particles during deposition is sufficient to overcome the energy barrier and form Al–O chemical bonds with O atoms on the Al<sub>2</sub>O<sub>3</sub> surface. Moreover, once these Al–O chemical bonds are formed, they can still be maintained after the Al film is melted. In this case, the molten aluminum can wet the ceramic at the just melting temperature. Therefore, although the sputtered Al film is solid, from the point of view of forming Al–O bonds, these Al films have "wetted" Al<sub>2</sub>O<sub>3</sub>. Therefore, despite the solid state, these Al films have "wetted" Al<sub>2</sub>O<sub>3</sub> regardless of whether the interface is liquid/solid or solid/solid.

## 5. Conclusions

In this paper, two kinds of  $Al_2O_3$  ceramic samples with and without Al film deposited were designed respectively. The influences of temperature and high kinetic energy sputtering particles on the wettability and interface strength of  $Al/Al_2O_3$  were studied by comparing the wetting behavior of molten aluminum on the surface of two samples. The effect of the kinetic energy of Al atoms on the wettability of  $Al/Al_2O_3$  was revealed.

1. For the sample without Al film deposited, molten aluminum does not wet  $Al_2O_3$  at 700 °C, the contact angle is 165°, and the interfacial shear strength is 25 MPa. With the increase in temperature, the contact angle decreases continuously, and the interface shear strength gradually increases. The contact angle decreases to a critical value of about 90° when the temperature increases to 1000 °C. With further increases in the temperature value of 1100 °C, the contact angle decreases to 80.3°. The wettability of molten aluminum to  $Al_2O_3$  ceramics changes from non-wetting to wetting. The shear strength of the joint gradually increases to 115 MPa at 900 °C, and then the increase slowed down.

2. The sample deposited with Al film is wetted by molten aluminum at 700  $^{\circ}$ C, and the contact angle is only 12 $^{\circ}$ . The interface shear strength is as high as about 120 MPa and is less affected by temperature. The shear fracture of the brazed joint occurs in the brazed seam of Al metal.

3. The high energy generated by either the temperature increase or the particle sputtering enables the Al atoms to overcome the energy barrier to form Al–O bonds with the O atoms on the  $Al_2O_3$  ceramic surface. The formation of Al–O chemical bonds is the fundamental reason for the improvement of  $Al/Al_2O_3$  wettability.

**Author Contributions:** Formal analysis, methodology, data curation, writing—original preparation, Y.L.; supervision, methodology, H.S.; investigation, writing—review and editing, B.M.; resources, validation X.G.; conceptualization, validation, R.L.; methodology and project administration, G.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Natural Science Foundation of China (No. 52002240).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

**Data Availability Statement:** The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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