

Changes in the Surface Characteristics of Gas-atomized Pure Aluminum Powder during Vacuum Degassing

Michiaki Yamasaki^a, Yoshihito Kawamura^b

¹Department of Materials Science, Kumamoto University, 2-39-1 Kurokami, Kumamoto 860-8555, Japan
^ayamasaki@gpo.kumamoto-u.ac.jp, ^brivervil@gpo.kumamoto-u.ac.jp

Abstract

Vacuum degassing is essential in the preparation of RS P/M aluminum alloys to remove adsorbates and for the decomposition of hydrated- Al_2O_3 on the powder surface. Changes in the surface characteristics during vacuum degassing were investigated by X-ray photoelectron spectroscopy and temperature-programmed desorption measurement. Hydrated- Al_2O_3 decomposition to crystalline- Al_2O_3 and hydrogen desorption on the surface of argon gas-atomized aluminum powder occurred at 623 K and 725 K, respectively. This temperature difference suggests that the reaction converting hydrated- Al_2O_3 to crystalline- Al_2O_3 during vacuum degassing should be divided into the two reactions " $2Al + Al_2O_3 \cdot 3H_2O \rightarrow 2Al_2O_3 + 6H_{surf}$ " and " $6H_{surf} \rightarrow 3H_2$ ".

Keywords : Aluminum, Rapid solidification, Powder metallurgy, Vacuum degassing, X-ray photoelectron spectroscopy.

1. Introduction

For several decades, new types of rapidly solidified powder metallurgy (RS P/M) Al alloys have been considered for a wide range of engineering applications because of their high specific strength. Powder sintering and hot pressing have been widely used as fabrication techniques for the P/M processing of bulk materials. However, consolidation techniques such as extrusion (rather than sintering) should be adopted when dealing with rapidly solidified alloys or amorphous alloy powders to prevent decomposition of the rapidly quenched phases during high temperature heat treatment. Although extrusion techniques simultaneously achieve dense compaction and the formation of metal-to-metal bonding between powder particles, vacuum degassing should be carried out before extrusion. There are two reasons why vacuum degassing is used prior to extrusion. The first is the removal of gases and water adsorbed from the powder-oxide layer. The second reason is the decomposition of ductile hydrated-alumina and/or aluminum hydroxide into brittle crystalline-alumina during vacuum degassing^[1]. The vacuum degassing process has a strong influence on the bonding of powder particles during their consolidation and on the strength of the extruded alloys. Excess degassing leads to decomposition of the rapidly quenched phases. On the other hand, insufficient degassing interferes with consolidation because of the formation of viscous hydrated-alumina and/or aluminum hydroxide. As mentioned above, the degassing process influences the performance of a P/M material both during manufacture and during use at high temperatures. Therefore, it is important to clarify the effect of vacuum degassing on the surface characteristics of gas-atomized Al alloy powders. In this study, we should consider the

effects of vacuum degassing on the decomposition of hydrated Al_2O_3 and the removal of adsorbed gases.

2. Experimental and Results

Rapidly solidified Al (99.99 %) metal powders with a mean particle diameter of about 25 μ m were formed from master alloy ingots by high-pressure argon gas atomization. In order to prevent the oxidation of these powders, all the processes were performed in a closed chamber and glove box, in which oxygen and moisture contents in the argon atmosphere were maintained at less than 0.5 ppm by a gas-purifier^[2]. The airtight sample tube taken out of the glove box was connected to the TPD measurement apparatus. Prior to TPD measurements, sample powders were exposed for 24 hours at room temperature to high purity argon gas with less than 0.5 ppm oxygen and moisture (clean powder), dry air with less than 20 ppm of moisture, or humid air with more than 40% moisture content. The Al powders were heated at a constant rate of 5 K min^{-1} in a vacuum of 10^{-3} Pa for TPD measurements. The gases desorbed from the powder surface were continuously analyzed by a quadrupole mass spectrometer. XPS of the surface region of the RS ribbon was carried out with a VG Scientific Sigma Probe at high temperatures of 323 to 873 K.

Figure 1 shows the H_2 TPD spectra for Al metal powders. H_2 desorption from the powders exposed to humid air began to increase sharply at 620 K and 660 K and peaked at 725 K. In contrast, no H_2 desorption was observed from the powders exposed to dry air or high purity argon.

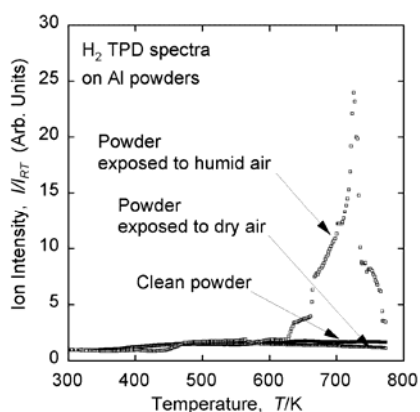


Fig. 1. H₂ TPD spectra for the aluminum metal powders exposed to humid air, dry air, or high purity argon gas.

Figure 2 shows the changes in the $[O^{2-}] / ([O^{2-}] + [H_2O] + [OH])$ atomic ratio as a function of degassing temperature. The ratio of $[O^{2-}] / ([O^{2-}] + [H_2O] + [OH])$ and increased with increasing degassing temperature. In this case, the XPS results suggest that the transformation from amorphous trihydrated-Al₂O₃ to crystalline γ -Al₂O₃ occurred at 623 K.

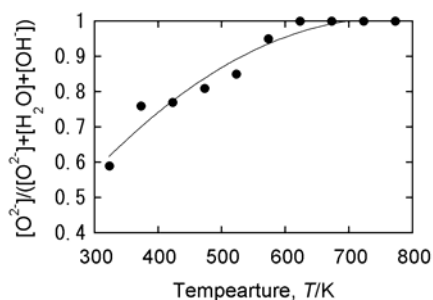
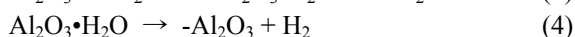
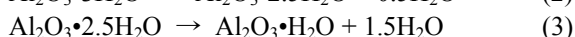
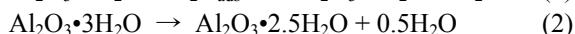
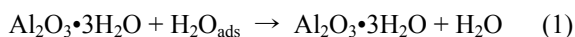
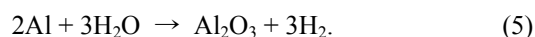


Fig. 2. Change in the $[O^{2-}]/([O^{2-}]+[H_2O]+[OH])$ atomic ratio as a function of degassing temperature.

Many previous studies have been carried out on the degassing of Al alloy powders. According to some past and more recent studies on air-atomized Al alloy powders, H₂O evolves at elevated temperatures as a result of the following reactions^[3],

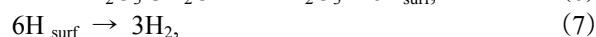


H₂O liberation occurs at a wide range of temperatures. When H₂O vapor comes into contact with Al, the following reactions take place, which in the principle source of H₂ evolution,



The undecomposed hydrated aluminum oxide layer obstructs the outward diffusion of Al³⁺ through the aluminum oxide layer. Accordingly, reaction eq. (5) does not occur below 620 K, resulting in no hydrogen desorption at low temperature of around 473 K.

The XPS results indicate that the hydrated-Al₂O₃ decomposition to crystalline-Al₂O₃ expressed in equation (5) occurs at 623 K. The hydrated-amorphous-Al₂O₃ decomposition temperature differs from the H₂ desorption temperature^[4]. These results suggest that the decomposition reaction of hydrated-Al₂O₃ expressed in eq. (5) should be divided into the two reaction eqs. (6) and (7) as follows:



where H_{surf} means a chemisorbed hydrogen atom in the surface layer region. The oxidation reaction of Al metal by water of hydration expressed in eq. (6) occurs at 623 K, while desorption of chemisorbed hydrogen from the surface expressed in eq. (7) occurs around 725 K.

3. Summary

The two reasons for vacuum degassing of Al alloy powders are the decomposition of ductile hydrated-Al₂O₃ to brittle crystalline-Al₂O₃ and desorption of the adsorbed gas species. The decomposition and desorption did not occur at the same temperature. The decomposition was completed at 623 K, while desorption of chemisorbed hydrogen occurred at a much higher temperature [5]. The optimum degassing temperature for Al powder consolidation during manufacture is 623 K. However, for use at high temperatures, a degassing temperature of more than 673 K should be considered to prevent blistering due to H₂ desorption in the final Al P/M alloy products.

4. References

1. M. Yamasaki and Y. Kawamura: Mater. Trans. **45**[4] (2004) 1335-1338.
2. Y. Kawamura, A. Inoue and T. Masumoto: Scr. Metall. **29** (1993) 25-30.
3. A. I. Litventsev and L. A. Arbuzova: Soviet Powder Metall. Met. Ceram. **49** (1967) 1-13.
4. M. Yamasaki and Y. Kawamura: Mater. Trans. **47**[8] (2006) in press.