Degassing of Aluminum and Aluminum Alloy Powders as Studied by Gas Chromatography

Ryuzo Watanabe¹,², Duk Sun Choi²,³, and Akira Kawasaki³

Department of Materials Processing, Tohoku University, Sendai, 980-8579 Japan
¹Now at Tohoku Polytechnic College, Aomori
171-2, Kitsuneno, Goshogawara, Aomori, 037-0002 Japan
Tel: +81-173-37-3201  Fax: +81-173-37-3203
²Now at Dong Sung Precision Co., LTD.
a, r-watanabe@aomori-pc.ac.jp,
c, kawasaki@material.tohoku.ac.jp

Abstract

Gas release behavior from aluminum and Al 7075 alloy powders during heating in argon was investigated by in-situ gas chromatography. Water vapor, hydrogen, carbon mono-oxide were detected as individual evolution spectra against heating temperature and time. The mechanisms of water and hydrogen evolutions were studied in detail for the determination of effective degassing condition. Magnesium in the alloy powder was found to lower the hydrogen evolution temperature to enhance overall hydrogen release.

Keywords: aluminum powder, gas release, gas chromatography, aluminum hydrate, excessively occluded hydrogen

1. Introduction

Sintered aluminum alloys are expected to be used as high specific strength materials for various machine applications. However, it is well known that gases discharged during heat-treatment, hydrogen in particular, result in an embrittlement of these materials, and degassing is indispensable before heat treatment [1][2]. Hydrogen dissolves well within molten aluminum, and due to a large difference in the solubility between those in solid and in liquid (the solubility ratio is 20:1), excess hydrogen precipitates during solidification. This produces product defects, such as inner porosity [2] and a surface swelling. Furthermore, dissolved hydrogen reacts with oxide impurities in aluminum to form H₂O, which preferentially precipitates on grain boundaries to form pores. These hydrogen pores will cause cracking at the grain boundaries and result in a grain boundary embrittlement. It seems that we need more information on the gas release from aluminum powders during degassing on a quantitative basis to discuss more in detail the degassing mechanism. In this study, we employed gas chromatography that identifies gas species and measures gas quantity, and traced the gas release behavior during heating of aluminum and aluminum 7075 alloy powder.

2. Experimental and Results

Sample powders were atomized aluminum and aluminum 7075 alloy powders containing 2.5 mass% Mg. The powders were heated in argon flow up to 500 °C at a heating rate of 2°C/min, kept at the maximum temperature for 1 h and furnace-cooled. Gas release behaviors during heating runs were followed by gas chromatography. The gas analysis system is composed of gas supplying part, heating furnace, TCD gas chromatograph with an automatic gas sampler, and a peak analyzer. Two kinds of absorbents, molecular sieve and porous polymer beads (Porapac N), were used for the two different gas groups. Molecular sieve 5A was used to detect hydrogen and carbon monoxide. Porapac N was used for argon, carbon dioxide and water vapor. The peak of carbon dioxide was fully covered with that of argon, and its evolution behavior was not discussed in the present paper. The peak of carbon dioxide can be determined by using helium as heating gas, which is the carrier gas in TCD gas chromatograph. This will be described elsewhere. The released gas quantity was expressed by TCD signals in microvolt second, and plotted against heating temperature and holding time. The blank test was conducted for each of the heating runs and the blank values were subtracted from those of the individual runs.

Figures 1 and 2 show the gas release behaviors for aluminum and aluminum alloy powders, respectively, in which release behaviors of water vapor, hydrogen, carbon monoxide are shown. Water vapor is released in the temperature range of 100 to 400°C with two peaks at around 100°C and 200-250°C, and hydrogen from 200 to 500°C, and carbon monoxide from 300 to 500°C. Comparing the gas release behaviors between aluminum and aluminum alloy the peak spectrum is broader in
aluminum alloy than in aluminum. Water vapor release will be attributed to the desorption of physically adsorbed water molecules for the first peak and to the decomposition of hydroxylaluminum attached on the particle surfaces for the second peak. **Figure 3** shows a comparison of the hydrogen release between aluminum and aluminum alloy powders. A clear difference can be seen in the diagram between two powders. The alloy powder starts to release hydrogen at lower temperature than non-alloyed powder. The released quantity is also much greater in the alloy powder. Hydrogen is the only gas that dissolves into solid aluminum but with very small quantity of 0.04 cm$^3/(100gAl)$. The sample powder was produced by atomization and the dissolved hydrogen in the melt is considered to be remaining supersaturated in the solid particles. The supersaturated hydrogen will diffuse out from the particles during heating. This will contribute to form the evolution peak. The second source of hydrogen will be provided by the reaction of aluminum and $H_2O$, according to the following reaction. $2Al + 3 H_2O = Al_2O_3 + 3 H_2$ ↑ (1). For the present alloy powder that contains 2.5% magnesium we can also postulate the following reaction. $Mg + H_2O = MgO + H_2$ ↑ (2). We know that magnesium is easier to be oxidized than aluminum, and the reaction (2) will take place prior to (1). Therefore the hydrogen evolution peak of the alloy powder will be formed by the reaction (2), the excessively occluded hydrogen and the reaction (1). The first peak would be originated from the reaction (2), while the second peak would be formed by the two sorts of contribution; one is from the reaction (1) and the other is from the excessively occluded hydrogen. The latter two contributions can not be separated in the present observation.

3. Summary

The water release is attributed to the desorption of adsorbed water on the particle surfaces and the decomposition of aluminum hydroxide. The hydrogen evolution is composed of two contributions: the release of the reaction product of aluminum and water and the excessively dissolved hydrogen within particles. The hydrogen evolution for the alloy powder is enhanced in the presence of magnesium which reacts with hydrated water.

4. References