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Original Research Paper

Efficiency of calcium vapor tunnels on non-contact deoxidation of irregular titanium powder

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ABSTRACT

This study developed a new process for the economical deoxidation of titanium powder in order to solve the problems associated with the previous deoxidation process. In the experiment, titanium powder with an irregular particle shape was used as the raw material with an oxygen concentration of 2100 ppm. The amount of Ca was reduced by 50% as compared to that used in a previous work and placed in an improved apparatus at a weight ratio of 2:1 (Ti:Ca). This modified deoxidation process decreased the oxygen concentration of the titanium powder to 805 ppm at 1273 K, leading to a ~61.7% reduction of oxygen compared to the raw titanium powder. Low-oxygen irregular titanium powder can be produced at a low cost and high efficiency by applying a non-contact deoxidation process using Ca vapor.

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1. Introduction

Recently, the use of powder metallic materials has increased owing to additive 3D-printing technologies [1–3]. In particular, 3D-printing technology has been attracting more attention because it can overcome the difficulties associated with machining high melting-point metals. Among the powders used in metallic materials, the demand for titanium powder has been increasing as it is highly compatible with the human body, and powders with the various shapes can be prepared through 3D-printing [4,5]. Early 3D-printing technology utilized spherical powders because most of the methods required powders with high fluidity. However, the recent widespread use of a bed-type 3D-printing technology allows for irregular powders with low fluidity to be used [6,7]. In general, titanium powder is classified as into spherical and irregular powders depending on its shape. Spherical powders have many advantages, including high fluidity and low oxygen concentration, but they suffer from a high production cost. The price of spherical titanium powder is several times higher than that of irregular titanium powder even for the same particle size and purity. Therefore, the powder metallurgy industry has attempted to reduce the price of products by using irregular titanium powder as the raw material. However, the high oxygen concentration of irregular titanium powder causes many problems in 3D-printing. High oxygen concentrations would interrupt the diffusion of inter-powder, leading to lower density of the final product [8,9]. Therefore, research on the deoxidation of titanium powder has been actively conducted to solve this problem [10–12].

In general, titanium powder is deoxidized to reduce TiO_2 into CaO, MgO, or Al_2O_3 by respectively reacting it with Ca, Mg, or Al, which have higher oxygen affinity than titanium [11]. Higher reaction temperatures also accelerate this deoxidation. However, a molten deoxidant with a significantly lower melting temperature than that of titanium is formed, thereby making it difficult to obtain pure titanium powder. To solve this problem, a previous study separated the deoxidant and titanium powder by using only Ca vapor to deoxidize the titanium powder [13]. The irregular titanium powder was deoxidized to an oxygen concentration of ~820 ppm. This irregular titanium powder had the lowest oxygen concentration among the powders typically used in the industry. However, this technique consumed a considerable amount of Ca, which is expensive, and hence was not economically viable. Considering that pure Ca metal is more expensive than titanium powder, efforts should be made to reduce the consumption of Ca while maintaining the level of deoxidation

In this study, we investigated the oxygen concentration of irregular and spherical titanium powders and maximized the reaction with titanium powder by setting up the Ca vapor tunnel in the non-contact deoxidation process, thereby reducing the amount of Ca expended.

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2. Experiments

The titanium powder used in this study had irregular polygon-like particles having an average size of 115 μm, with 100 mesh and an initial oxygen concentration of 2100 ppm. The titanium powder was a product of KISWEL and was obtained by a hydrogenation-dehydrogenation (HDH) process. Ca (JUNSEI Chemical Company) used as the deoxidant had a purity of 99.5% and an average particle size of 5 mm or less. The titanium powder and Ca were filled in the improved apparatus at a ratio of 2:1. The deoxidation was initiated under high vacuum (5×10^{-5} torr) with a heating rate of 10 °C/min. Once the ramping process was complete, the temperature was held constant for 1 h at each temperature in the range of 973–1273 K. The experiment was classified into two types based on the melting temperature of Ca: surface contact-type of deoxidation, in which titanium powder and Ca were in direct contact at 973 and 1073 K; and non-contact-type deoxidation, where only Ca vapor was used at 1173 and 1273 K, by separating Ca and titanium powder. After deoxidation, CaO obtained from the titanium powder was washed repeatedly with water and 10% hydrochloric acid, and then dried at 60 °C for 2 h in a vacuum oven. Scanning electron microscopy (SEM, Jeol, JSM-6380LA) was conducted to observe the macroscopic surface conditions of the titanium powder. The oxygen concentration of the deoxidized titanium was measured using a gas analyzer (ELTRA ON-900). The gas analysis was carried out three times, and the oxygen concentration was calculated as an average value. According to our analyses on the oxygen concentration, the error range for the gas analyzer (ELTRA ON-900) was ±40 ppm. X-ray diffraction (XRD) analysis (RIGAKU, MAX-2500) was carried out to confirm the phase changes and the presence of the remaining Ca or Ca compounds.

3. Results and discussion

Fig. 1(a) and (b) show the SEM images of titanium powder with similar particle size of 100 mesh distribution. The irregular powder had much more inconstant edges, which are known to form oxides. Fig. 1(c) shows the oxygen concentrations according to the particle shape and particle size of titanium powder produced by various suppliers and currently commercially available. The purity of all the samples was 99.9%. Each maker produces and sells spherical and irregular titanium powder having various particle sizes. Irregular powder is mainly produced by the HDH method, and spherical powder is produced through the atomization process. Owing to the differences in the manufacturing process and raw materials used, the price differs depending on the shape, even for powders produced by the same supplier. The irregular titanium powder we investigated had a much higher oxygen concentration than the spherical powder. In addition, the oxygen concentration of the titanium powder was inversely proportional to the particle size. The oxygen concentration of the titanium powder can be expressed by the following equation [14].

$$O_{total} = O_{internal} + O_{surface} = O_{internal} + a \times t \times P_{TiO2} \times O_{TiO2}$$

In the formula, a is the specific surface area of the titanium powder, t is the thickness of the oxide layer, P_{TiO2} is the density of TiO_2, and O_{TiO2} is the weight percentage of TiO_2. Since $O_{internal}$, t, P_{TiO2}, and O_{TiO2} do not change during the pulverization process, the largest influence is due to the specific surface area, and the equation is reduced as follows:

$$O_{total} = O_{internal} + 1.69 \times 10^{8} ta$$

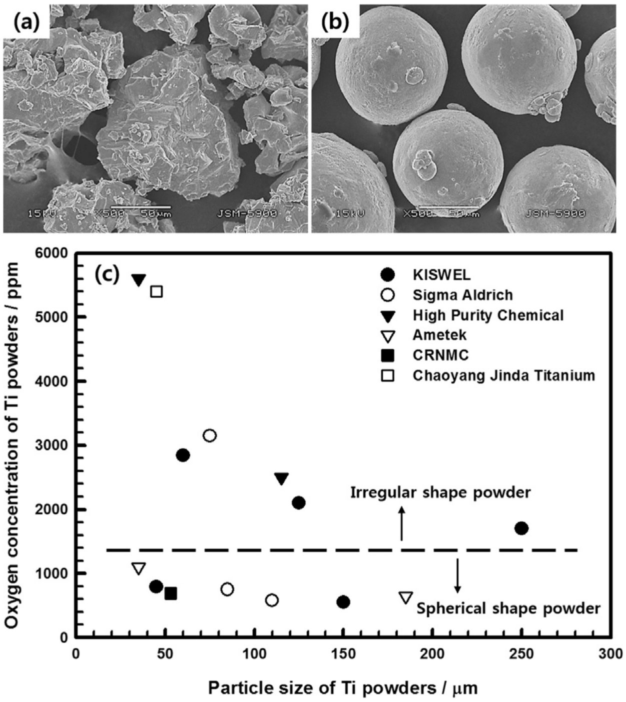

Fig. 1. SEM images of (a) the irregular and (b) spherical titanium powders. (c) Oxygen concentration according to the shape and particle size of titanium powder produced by various makers and currently available in the market.

However, the irregular titanium powder was prepared by the HDH process, and the oxide layer was irradiated with about 10 nm [15]. Assuming that titanium powder is a perfect spherical shape, the theoretical specific surface areas of titanium powders with 45 μm and 115 μm are 2.96 m^2/g and 1.15 m^2/g, respectively. It is found that titanium powder with a smaller particle size has a much larger specific surface area. Therefore, if the oxygen concentration of irregular titanium powder with 100 mesh distribution can be decreased to below 1000 ppm, the resulting powder can be fully utilized in the 3D-printing industry. This demand has led to the development of the deoxidation technology for titanium powder using Ca vapor. However, this technique is not economically viable because of the large consumption of Ca. This technique also leads to difficulties in the recovery of pure titanium powder. Therefore, we improved the deoxidation process to overcome these problems. Fig. 2(a) shows the internal schematic of the improved deoxidation apparatus. The Ca vapor in the lower part moves smoothly through the tunnel installed inside the deoxidation apparatus. Even though the amount of Ca is reduced in this case, a greater amount of Ca vapor can flow through the tunnel, thereby accelerating the reaction with the titanium powder. In addition, it is expected that Ca and its reactants arising from the absence of the tunnel in the existing device and cumulated on the bottom of the micro-sieve are reduced, resulting in the easier recovery of the powder.

Fig. 3 shows the oxygen concentration of the titanium powder obtained by the improved deoxidation process. The oxygen concentration of the initial raw (titanium powder) was measured as 2100 ppm. The titanium powder was deoxidized Ca through a surface contact reaction at 973 or 1073 K, which reduced the oxygen concentration to 1390 or 1040 ppm, respectively. Then, deoxidation was performed at 1173 K or 1273 K using Ca vapor (non-contact) because deoxidation of titanium powder using a contact method at temperatures above 1108 K creates molten Ca, complicating the recovery of titanium powder. The deoxidations performed at 1173 and 1273 K using the improved apparatus reduced the oxygen concentrations to 840 and 805 ppm, respectively. The oxygen concentration was reduced by ~61.7% compared to the initial concentration in the raw titanium

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