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Synthesis of nanosized alumina powders by pulsed wire discharge in air flow atmosphere

Satoru Ishihara *, Hisayuki Suematsu, Tadachika Nakayama, Tsuneo Suzuki, Koichi Niihara

Extreme Energy-Density Research Institute, Nagaoka University of Technology, 1603-1 Kamitomioka, Nagaoka, Niigata 940-2188, Japan

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Abstract

Pulsed wire discharge (PWD) is a method to form nanosized powders which is based on evaporation of thin metal wires by applied large electric current. Nanosized oxide powders can be synthesized by PWD in oxygen atmosphere. Since only small amount of powders can be synthesized by single discharge to a piece of wire, the number of discharges has to be increased for mass production of nanosized powders. Then, PWD equipments with wire feeding and gas flow atmosphere systems were developed for mass production. However, effects of gas flow on formed powders have never been investigated. In this study, PWD in air flow atmospheres were tried to synthesize alumina powders from industrial viewpoints. The effects of air flow rate on the oxidation phenomena and the synthesized powders have been investigated. PWD of pure Al wires with diameters of ϕ 0.3–0.5 mm were examined in air flow rates of 15 L/min and 33 L/min as well as 0 L/min (closed atmosphere condition). The synthesized powders at all experimental conditions were dominantly composed of γ-Al_2O_3 and δ-Al_2O_3 phases. The δ/γ ratios of the Al_2O_3 phases increased with increasing air flow rate and wire diameter. Besides them, small amount of metallic Al phase was remained in the powders synthesized by PWD in the air flow atmosphere of 33 L/min. It is concluded that appropriate air flow rate should be adopted for mass production of alumina powders in order to achieve enough collection of particles and avoid mingling of metallic Al phase.

Keywords: A. Powders: gas phase reaction; D. Al_2O_3

1. Introduction

Various kinds of processes have been developed to form nanosized powders whose scales in the range of 1–100 nm, since the size effect of decreasing diameter and the consequent increasing in the specific surface area bring some unique functions and properties. These processes can be classified into liquid phase methods and gas phase methods according to the principle of particle formation. The pulsed wire discharge (PWD) method was developed to form nanosized powders, which is categorized in the gas phase methods [1]. The method yields nanosized particles by pulsed electric current passing through metal wires which are then quickly evaporated. High efficiency of the direct energy conversion from electricity to heat is one of the principal advantages against the other gas phase methods. In cases of PWD performed in inert gas atmosphere, nanosized metallic powders of same composition as the discharged wire are formed from the metal vapor by simple cooling. In cases that oxygen or air is used as the atmosphere, the evaporated metal is oxidized and results in formation of nanosized particles of oxide such as alumina and titania [2–8].

Only small amount of powders can be synthesized by single discharge to a piece of wire. For mass production of nanosized powders by PWD, the number of discharges has to be increased by additional systems such as wire feeders to supply fresh wires between electrodes after a discharge, and an automatic controller to synchronize the wire feeder with the discharge [9,10]. In these developed equipments, continuous gas flow is operated as the atmosphere condition so that the formed particles do not accumulate on the bottom of the discharge chamber but are collected on a filter. However, effects of gas flow on formed particles have never been investigated. Especially in cases of synthesizing oxide particles, gas flow will influence not only the cooling of vapor but the oxidation phenomena. Though closed oxygen atmosphere was usually

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* Corresponding author. Tel.: +81 258 47 9893; fax: +81 258 47 9890.
E-mail address: ishihara@etigo.nagaokaut.ac.jp (S. Ishihara).

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used for most of previous investigation to synthesize oxide powders by PWD [2–8], air is more suitable atmosphere than oxygen for mass production of alumina particles as industrial application taking into account of the cost and safety. In this study, PWD in air flow atmospheres were tried to synthesize alumina powders from industrial viewpoints. The effects of air flow rate on the oxidation phenomena and the synthesized powders have been investigated.

2. Experimental procedure

Commercially available pure Al wires (The Nilaco Co., Tokyo) in the diameters of ϕ 0.3 mm, ϕ 0.4 mm, and ϕ 0.5 mm were used as the raw materials to be discharged. A schematic diagram of the PWD apparatus used in this study is shown in Fig. 1. The PWD experiments were carried out according to the following procedure. A piece of pure Al wire was set between the electrodes (the nominal span length: 25 mm) in the chamber for each experiment of discharge. The volume of the chamber is 22 L, which is almost same as the molar volume of gases at the standard temperature and pressure. The air flow atmosphere in the chamber was formed by regulated air evacuation, which the gas inlet was released and the gate valve was fully or partially opened. The air flow rates were evaluated by a gas flow meter as 33 L/min and 15 L/min for the fully and partially opened gate valve conditions, respectively. Experiments in closed atmosphere were also carried out for comparison as the air flow rate of 0 L/min at the state of which the gas inlet and the gate valve were closed. The capacitor bank was charged from the high-voltage DC power supply. Pulsed large current from the capacitors was applied to the set wire by closing the spark gap. After the resultant wire evaporation, the formed particles in the chamber were evacuated and collected on a membrane filter of 0.1 μm pore size. The collected powders were analyzed by X-ray diffraction (XRD; Rigaku, RINT2000, Cu Kα, 50 kV, 300 mA) and transmission electron microscopy (TEM; JEOL, JEM-2000FM). The size distributions of particles synthesized at the respective PWD conditions were evaluated by an image analysis software from the TEM images.

Table 1
Experimental conditions.

Wire diameter, d_w	ϕ 0.3 mm	ϕ 0.4 mm	ϕ 0.5 mm
Vaporization energy, E_v	67.3 J	120 J	187 J
Air flow rate, R_{af}	0 L/min (closed), 15 L/min, 33 L/min		
Capacitance, C	20 μF		
Charging voltage, V_c	6 kV		
Charged energy, E_c	360 J		
Relative energy, $K = E_c/E_v$	5.4	3.0	1.9

The experimental conditions adopted in this study are summarized in Table 1. The vaporization energy (E_v) of pure Al wire was calculated from the enthalpy change to vaporization at the boiling point [11], and the nominal volume of wires. The charged energy, E_c can be calculated by the equation;

$$E_c = \left(\frac{1}{2}\right) C V_c^2 \tag{1}$$

where C is the capacitance of the capacitor bank and V_c is the charging voltage, respectively. To evaluate the effect of discharge in PWD, relative energy, K which is defined as the ratio of the charged energy of the capacitor E_c to the vaporization energy of the wires E_v;

$$K = \frac{E_c}{E_v} \tag{2}$$

is often used as an important index. In this study, the value of relative energy, K is in the range between 1.9 and 5.4, which means enough energy can be applied to vaporize each Al wire from the charged capacitor.

In these PWD experiments, the voltage between the electrodes and the current of the discharge circuit during each discharge were measured with the digitized oscilloscope using two high-voltage probes and the current transformer. The temperatures of the vapor during the wire explosion were

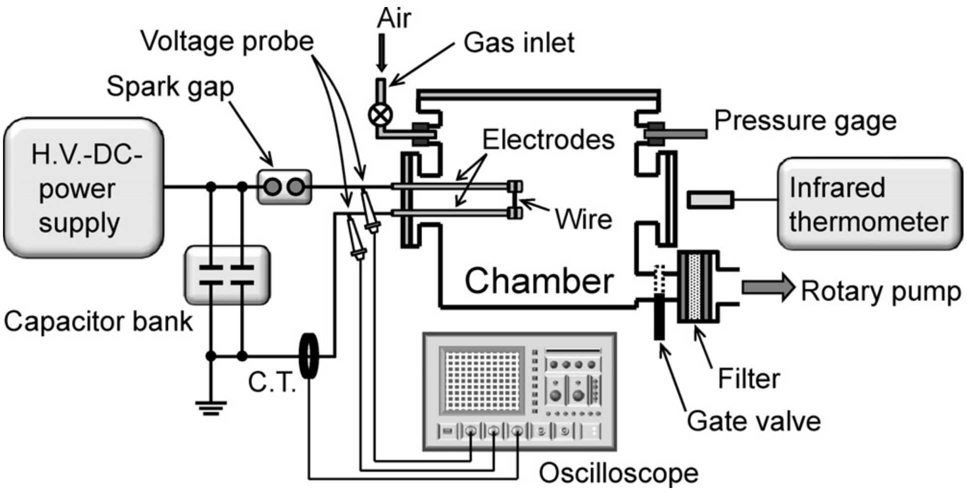

Fig. 1. Schematic diagram of the PWD apparatus used in this study. The gas inlet was released and the gate valve was fully or partially opened for the air flow atmosphere.

Table 2
Specification of the high-speed infrared thermometer.

Measuring range	400–3000 °C
Spectral range	2.0–2.5 μm
Response time	10 μs
Measuring field	About ϕ 1 mm

measured with a high-speed infrared thermometer (Kleiber, KMGA 740-OL). Specification of the used infrared thermometer is shown in Table 2. Temperature measurement area was set at the center of the wire between the electrodes. The temperature measurements were carried out within the criteria predicted by a numerical simulation [12].

3. Results

White powders were obtained by the PWD experiments at all the experimental conditions in this study. Fig. 2 shows the XRD patterns of the powders synthesized by the PWD experiments of the ϕ 0.3 mm Al wire at the various conditions of air atmosphere. Most of the diffraction peaks can be identified as γ-Al_2O_3 and δ-Al_2O_3 phases. This result is similar to many reports on synthesizing alumina by the PWD method [2,3]. Whereas many of diffraction peaks with high intensity have possibility of diffraction by both the γ-Al_2O_3 and δ-Al_2O_3 phases, relative intensity of the γ- and δ-Al_2O_3 phases can be evaluated by comparison of the diffraction peaks at $2\theta = 37.6°$ and 36.5° as shown as the lines in Fig. 2, for γ- and δ-Al_2O_3 phases, respectively. In this study, the comparison of these diffraction peaks indicates that the relative peak height of the γ phase against the δ phase decreased with air flow rate. Small diffraction peaks by the α-Al_2O_3 phase are detected in the cases of closed and 33 L/min conditions in this study. In addition to these alumina phases, small amount of metallic Al phase was remained in the powders synthesized at the only condition in air flow rate of 33 L/min as shown as the peak on the dot line at the diffraction angle of $2\theta = 38.5°$ in Fig. 2, while no diffraction peak is detected at this diffraction angle in the cases of closed and 15 L/min conditions.

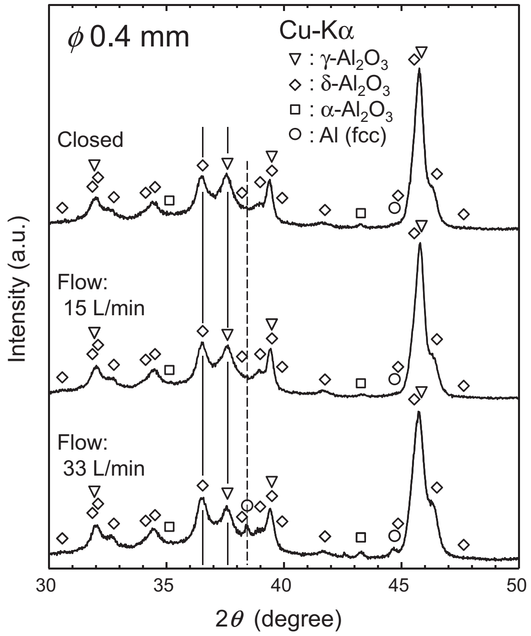

Fig. 3. XRD patterns of the powders synthesized by PWD of the ϕ 0.4 mm Al wire at the various conditions of air atmosphere.

The XRD patterns of the powders synthesized by the PWD experiments at the various conditions of air atmosphere for the Al wires of ϕ 0.4 mm and ϕ 0.5 mm are shown in Figs. 3 and 4,

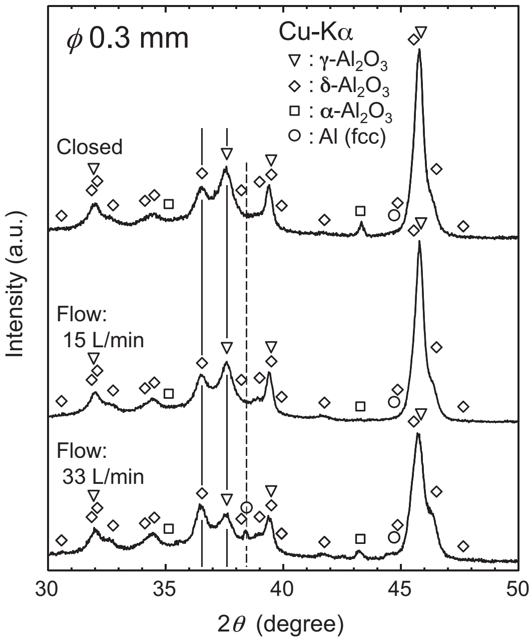

Fig. 2. XRD patterns of the powders synthesized by PWD of the ϕ 0.3 mm Al wire at the various conditions of air atmosphere.

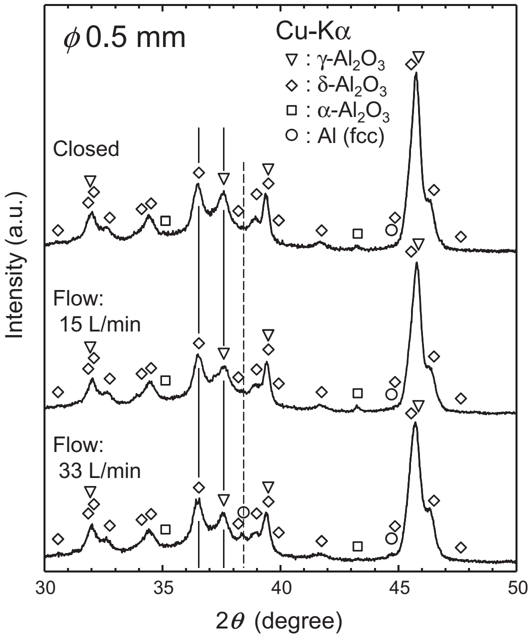

Fig. 4. XRD patterns of the powders synthesized by PWD of the ϕ 0.5 mm Al wire at the various conditions of air atmosphere.

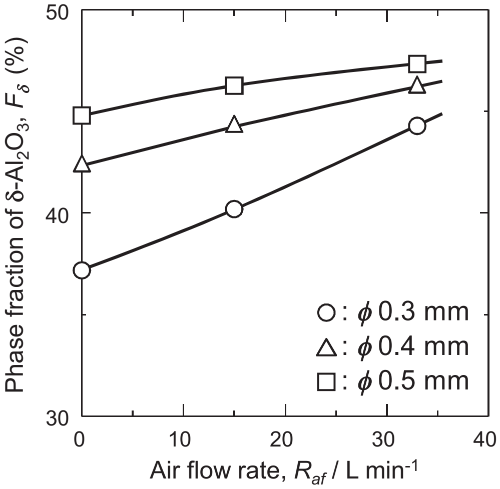

Fig. 5. Relationship between phase fraction of δ-phase in the synthesized powders and air flow rate.

respectively. These powders were mostly composed of γ-Al_2O_3 and δ-Al_2O_3 phases as same as the case of the ϕ 0.3 mm wire which is shown in Fig. 2. The comparison of these diffraction peaks indicates that the relative peak height of the γ phase against the δ phase decreased with air flow rate. Small amount of metallic Al phase was remained in the powders synthesized at the only condition in air flow atmospheres of 33 L/min at both the wire diameter conditions, while no diffraction peak is detected at the diffraction angle in the cases of closed and 15 L/min conditions. This tendency is same as those in the case of the ϕ 0.3 mm wire.

In order to analyze the δ/γ ratios of the Al_2O_3 phases in the obtained powders, the integrated intensity for the peaks at $2\theta = 37.6^\circ$ and 36.5° was evaluated for the γ- and δ-Al_2O_3 phases, respectively. As these results, the phase fraction of δ-Al_2O_3 phases in amount of the γ-and δ-Al_2O_3 phases is plotted in Fig. 5 as a function of air flow rate. The phase fraction of δ-Al_2O_3 phase in the obtained powders increased with air flow rate at the same wire conditions. The phase fraction of δ-Al_2O_3 phase increased with wire diameter at the same air flow rates.

Bright-field TEM images of the particles synthesized at the various conditions are shown in Fig. 6. The shape of these particles synthesized at all conditions is almost spherical. The diameters of the observed particles are in the range from single nm to 100 nm. The larger particles are observed in the images synthesized from the larger diameter in wires. The size distributions of these particles were evaluated by an image analysis from the respective TEM images. The results of the size distribution are shown in Fig. 7. The curves in the respective figures are fitted log-normal distribution to the experimental data. The median diameter of particles increases with the wire diameter in both closed and 33 L/min conditions. Such results were obtained in previously reported research about formation of metallic powders by PWD [13]. The particle distribution at the both the cases of atmosphere condition are almost similar at the respective wire diameter, although relatively finer particles become slightly more and relatively larger particles become slightly less in the cases of 33 L/min condition as compared with closed condition as shown as the dot lines in Fig. 7(d)–(f).

Fig. 8 shows the waveforms of the voltage between the electrodes, v, and the current of the discharge circuit, i, during the respective discharge. While the left side figure shows the dependence of wire diameter, the right side is for the effect of air flow. Each datum point of time, $t = 0$ was defined at the beginning of the first increase in current of the discharge circuit for respective waveforms. In all the results obtained at any experimental conditions, each voltage waveform has a peak and the current waveform has a drop at the same time. The voltage peak and current drop on the waveforms can be explained by an abrupt increasing in the resistance of the wire or between

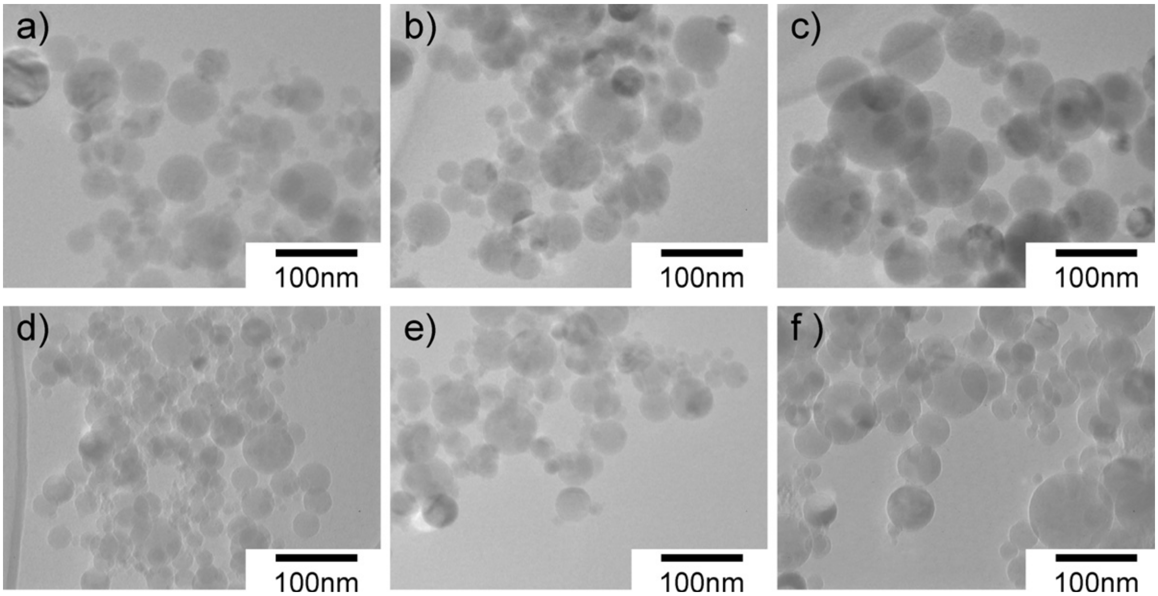

Fig. 6. Bright-field TEM images of the particles synthesized by PWD at the various conditions of wire diameter and atmosphere (wire diameters of (a) and (d): ϕ 0.3; (b) and (e): ϕ 0.4; (c) and (f): ϕ 0.5, and atmosphere of (a), (b), and (c): closed; (d), (e), and (f): air flow of 33 L/min).

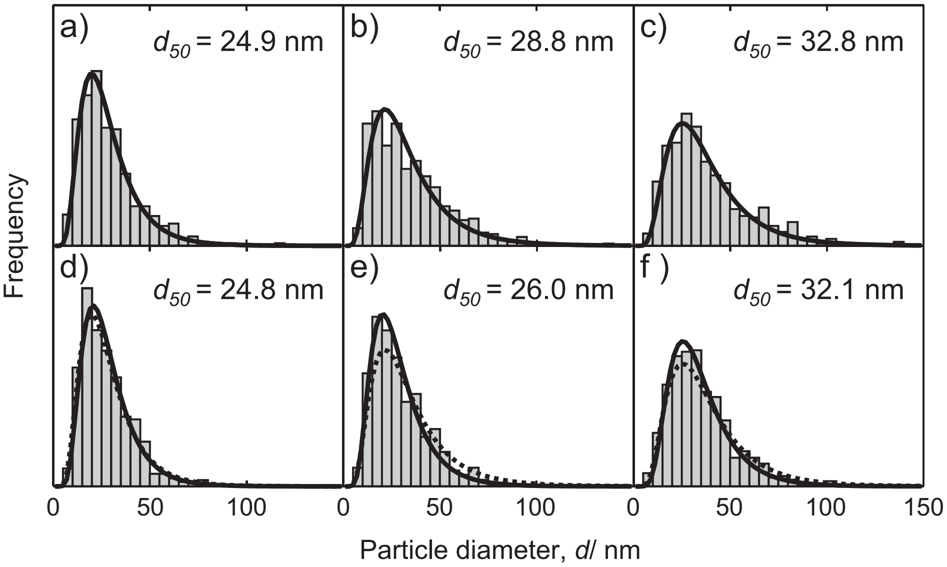

Fig. 7. Particle diameter distributions of the particles synthesized by PWD at the various conditions of wire diameter and atmosphere (wire diameters of (a) and (d): ϕ 0.3; (b) and (e): ϕ 0.4; (c) and (f): ϕ 0.5, and atmosphere of (a), (b), and (c): closed; (d), (e), and (f): air flow of 33 L/min). The dot lines in (d), (e), and (f) correspond to the distribution lines in (a), (b), and (c), respectively.

electrodes due to the wire vaporization [14]. After the vaporization and disappearance of the wire which composed a part of the electric circuit to discharge, the electric current can be continued by an arc discharge in the formed plasma and/or high-temperature vapor between the electrodes until the all charged energy in the capacitor bank is released. In the comparison among the results in dependence of wire diameter, Fig. 8 – left side part, the time at voltage peak and current drop

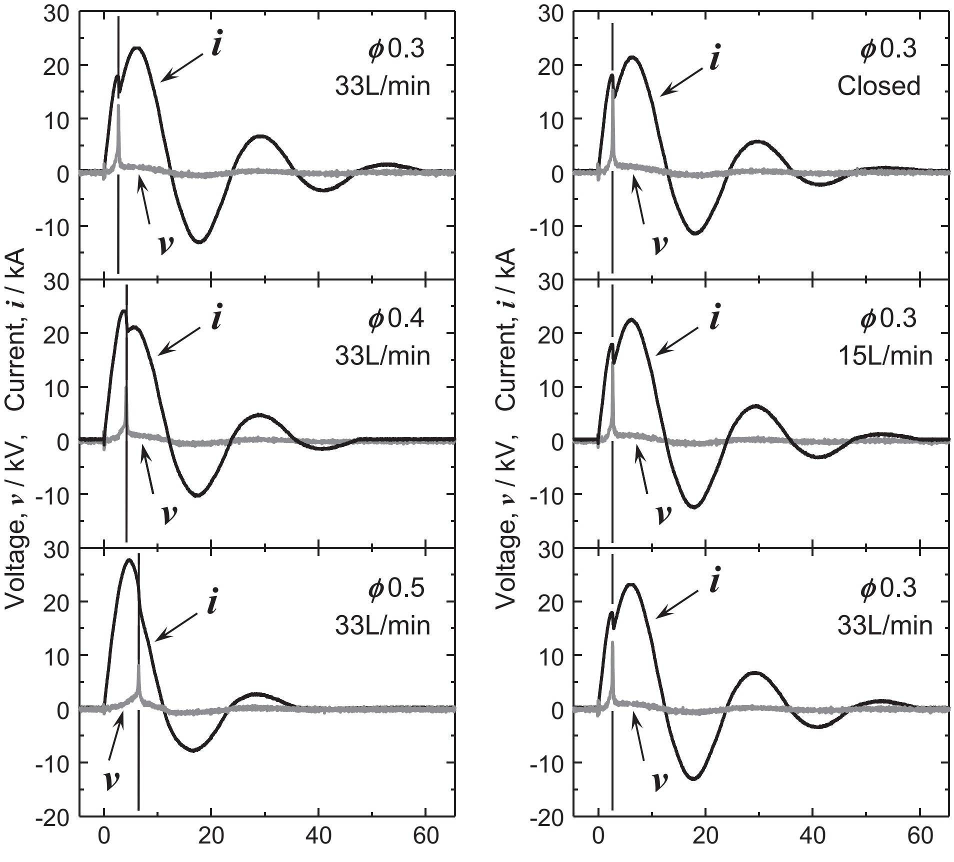

Fig. 8. Waveforms of voltage, v and current, i in PWD at the various conditions of wire diameter in air flow atmosphere of 33 L/min (left side), and at the various conditions of atmosphere (right side).

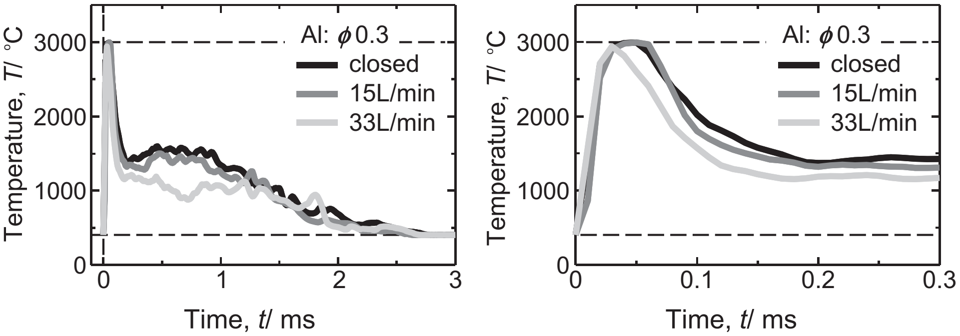

Fig. 9. Time evolution of temperature measurement by the high-speed infrared thermometer during the discharge in the PWD experiments of Al wires with the diameter of ϕ 0.3 mm at various air flow atmosphere conditions. The right side is enlarged at the time range of 0–0.3 ms.

were delayed with increasing the wire diameter. It can be explained by the larger vaporization energy and the consequent longer time to reach the vaporization in the larger wire diameter for same discharged electric energy. The waveforms of the voltage and the current at the different atmosphere conditions shown in Fig. 8 – right side part are almost similar, respectively. The voltage peak and current drop were detected at the almost same time of about 3 μs. These results on the similar voltage and current evolution at all the atmosphere conditions indicate that the discharge phenomena are hardly influenced by the atmosphere conditions.

Fig. 9 shows the time evolution of temperature measured by the high-speed infrared thermometer during the PWD experiments of Al wires with the diameter of ϕ 0.3 at various air flow atmosphere conditions. The right side part is enlarged in the time range of 0–0.3 ms. The measurement range of the thermometer is 400–3000 °C, and consequently the measured values are indicated as 400 °C and 3000 °C in the cases of actual temperatures below 400 °C and over 3000 °C, respectively. An abrupt increase in temperature over the boiling point of Al, 2519 °C and a following decrease were recognized by the measurement in each PWD at all the atmosphere conditions. In the cases of closed and 15 L/min, the increased temperature kept over 3000 °C up to time of about 0.06 ms, although the actual temperature cannot be measured by the thermometer over 3000 °C. This time range is in good agreement with the time that the current became zero on the results of the current measurements by the oscilloscope as shown in Fig. 8. It is concluded from these results that the heating is caused by the current through the wires up to the wire vaporization and the following arc discharge between the electrodes. In the cases of 33 L/min, the abrupt increase in temperature by the discharge reached 3000 °C and decreased up to about 1200 °C at slightly earlier time than the other air flow conditions.

In the cases of closed and 15 L/min, another temperature increase was found after the first decreasing at about 0.2 ms. Since the heating by electrical energy was completed at about 60 μs as mentioned above, it is consequently concluded that the second increase in temperature can be considered to be caused by oxidation of residual metallic aluminum after the first heating by electrical energy [15]. This second increase in temperature at the condition of 15 L/min is a little lower than that of the closed atmosphere. In the case at the condition of 33 L/min, such second increase in temperature was not clearly detected. These results mean that the second increase in temperature is suppressed by the air flow. However, temperatures over 400 °C after the second increase in temperature lasted up to 2.7 ms as same as the other atmosphere conditions.

4. Discussion

In this study, PWD experiments of pure Al wires with the diameter of ϕ 0.3–0.5 mm were tried in air flow atmospheres of 15 L/min and 33 L/min as well as the closed air atmosphere, in order to investigate the effects of air flow on the oxidation phenomena and the synthesized particles. The synthesized powders in all experimental conditions were dominantly composed of γ-Al_2O_3 and δ-Al_2O_3 phases. The Al_2O_3 ceramic system is known to exist in a number of metastable polymorphic forms such as γ, δ, θ, η, κ, χ, besides the thermodynamically stable phase of α which is often called corundum [16]. Alumina particles synthesized by most of gas phase methods have been reported to consist predominantly of γ-Al_2O_3 phase and partly of other metastable phases such as δ-Al_2O_3 and θ-Al_2O_3 phases, rather than the most stable α-Al_2O_3 form [17–22]. It is considered that formation of these metastable Al_2O_3 phases is common phenomena in nanosized powders synthesized by gas phase methods. The reason can be explained by the relative thermal stability change in nanosized powders, which is caused by effects of increased surface area [23].

As the effect of the wire diameter, the phase fraction of δ-Al_2O_3 increased with wire diameter at the same air flow rate and charging conditions. This result means that higher temperature phase of δ-Al_2O_3 against γ-Al_2O_3 is more formed by lower ratio of charging energy for mass of Al which depends on wire diameter. This tendency is same as the previous result, and can be explained as that the second increase in temperature becomes higher due to the spontaneous oxidation of residual metallic aluminum component though the first increase in temperature becomes lower in cases at lower ratio of charging energy against vaporization energy of wires [15]. The median diameters of the synthesized powders increased with the wire diameter. Similar results were obtained in previous research and explained as the effect of vapor density formed at each discharge [13,24]. Higher density of vapor is formed by large mass of Al from the wires at the discharge and higher density of clusters formed in the vapor results in formation of larger particles.

As the effects of the air flow atmosphere, the phase fraction of δ-Al_2O_3 increased with air flow rate at the same wire diameter and charging conditions. This result means that higher temperature phase of δ-Al_2O_3 is more formed by higher air flow rate. It is difficult to explain the reason since the measured temperatures in both the first and second temperature increase stage at the flow rate condition of 33 L/min were lower than those at the other atmosphere conditions. Although the maximum temperature at the second increase in temperature among the air flow conditions, period with temperatures over 400 °C after the second increase in temperature lasted up to 2.7 ms at all the air flow conditions. In the cases of higher charging energy which form lower fraction of δ-Al_2O_3 phase, the maximum temperature at the second increase stage was lower and the period at temperatures over 400 °C became shorter [15]. The different tendencies of period to decrease in temperatures up to 400 °C suggests that the second increase in temperature at higher flow rate in this result is not same as the cases at higher charging energy. As the direct effects of air flow in PWD, it is considered that the flow of air dilutes the density of Al vapor and supplies further oxygen to the Al vapor. Therefore, respective particles can be more self-heated by spontaneous oxidation enhanced by the additional oxygen which is supplied by the air flow. However, the Al vapor is diluted by the air flow and the temperature of the vapor might be apparently measured lower. It can be considered based on both the effects of air flow that higher phase fraction of δ-Al_2O_3 due to the spontaneous oxidation enhanced by the additional oxygen, although the measured temperature of the vapor is apparently low. As shown in Fig. 7, relatively finer particles become slightly more and relatively larger particles become slightly less in the cases of 33 L/min condition as compared with closed condition at the respective wire diameter. The reason can be explained also based on the dilution of Al vapor by the air flow, since particle diameters in PWD usually depend on density of vapor formed by discharge [13,24], as above mentioned in the discussion about the effect of wire diameter on the particle diameters. In the cases of air flow conditions, Al vapor formed by discharge is diluted by the air flow and consequently finer particles can more form as compared with the closed conditions. However, the effect by air flow is not so large to decrease significantly median diameters.

Besides γ-Al_2O_3 and δ-Al_2O_3 phases, small amount of metallic Al phase was remained in the powders synthesized by PWD in the air flow atmosphere of 33 L/min. This reason can be explained as that large air flow dilutes the vapor and a part of vapor is cooled to solid particles without being oxidized. Such mingling of metallic Al phase is not desirable for mass production of alumina particles. It is concluded that appropriate air flow rate should be adopted for mass production of alumina particles in order to achieve enough collection of particles and avoid mingling of metallic Al phase.

5. Conclusions

PWD in air flow atmosphere was tried to synthesize alumina powders from an industrial viewpoint in this study. The effects of air flow rate on the oxidation phenomena and the synthesized powders have been investigated. The synthesized powders in all experimental conditions were dominantly composed of γ-Al_2O_3 and δ-Al_2O_3 phases. The δ/γ ratios of the Al_2O_3 phases increased with increasing air flow rate and wire diameter. Besides them, small amount of metallic Al phase was remained in the powders synthesized by PWD in the air flow atmosphere of 33 L/min. It is concluded that appropriate air flow rate should be adopted for mass production of alumina particles in order to achieve enough collection of particles and avoid mingling of metallic Al phase.

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