Large-scale Synthesis of Functional Nanopowder using Modulated Induction Thermal Plasmas with Intermittent Feedstock Feeding

原料供給・電力の変調を伴う誘導熱プラズマを用いた 機能性ナノ粒子の大量生成

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A novel method was developed for large-scale synthesis of nanopowder using the pulse modulated induction thermal plasmas (PMITP) with time-controlled feeding of feedstock (TCFF). The PMITP produces repetitious relatively high-temperature and low-temperature fields, where the feedstock is fed intermittently synchronized with the coil current modulation. This method provides high-efficient complete evaporation of feedstock during the high-temperature field, and high efficient nucleation of nanoparticles in the successive low-temperature field. Use of this method produces Al doped TiO_2 nanopowder with a high production rate of 400 g/h at 20 kW as an example.

1. Introduction

Nanoparticles are anticipated as promising next generation elements to various applications such as in electronics, energy and environmental fields, etc [1]. The titanium dioxide (TiO₂) nanoparticles are ones of attractive nanoparticles widely used as photocatalyst materials under ultraviolet light because of its wide energy band gap. Attention has been also paid to TiO2 nanoparticles with metallic-ion doping as photocatalyst materials. This is because the impurity energy levels in the band gap improve their visible light absorption efficiency [2]. Another application of such metallic-ion doped TiO₂ particles is in biomedical field. It has been recently reported that Al-doped TiO₂ has a protein adsorption ability, which is effective to skincare for atopic dermatitis [3]. However, effective mass production methods have been not yet developed for such metal-doped TiO₂ nanopowder without any contamination.

We have developed a large-scale synthesis method of nanopowder using the pulse-modulated induction thermal plasmas (PMITP) with time-controlled feeding of feedstock (TCFF) method [4]. The PMITP has been developed by our group to control the temperature and reaction fields in thermal plasmas by the coil current modulation. In addition to this, a method was also developed for feedstock powder to be intermittently supplied to the thermal plasmas. Such the TCFF was combined with the PMITP for synthesis of large amounts of nanopowder.

This paper briefly introduces the developed PMITP-TCFF method for large-scale synthesis of nanopowder. Results concerning morphology from FE-SEM and total diffusive reflectivity are indicated for Al doped TiO_2 nanopowder and Fe-doepd TiO2 nanopowder synthesized by the PMITP-TCFF method.

2. Methodology of the PMITP-TCFF method

Figure 1 shows the schematic diagram of the PMITP-TCFF method. The PMITP can repetitively produce a higher temperature and high-reactive field during the on-time, and a lower temperature field during the off-time in the plasma torch. [4] Feedstock was injected into the PMITP intermittently and selectively only during the on-time with a high-speed valve. In this case, the injected feedstock to the PMITP was effectively

and completely evaporated in a high-temperature plasma during the on-time. On the other hand, injection of feedstock was stopped in the off-time to decrease the PMITP temperature and the evaporated material. Such rapid temperature decay may promote particle nucleation from evaporated feedstock in the PMITP. In addition, quenching gas was injected downstream of the torch in the radial direction. This quenching gas injection is also effective to prevent from particle growth. The above processes including effective vaporization of feedstock and effective cooling down of evaporated material may make it possible to synthesize a large amount of nanopowders with a high production rate.

3. Properties of synthesized nanopowder

Figure 2 shows the FE-SEM images of synthesized Al-doped TiO₂ nanopowder using the Ar-O₂ PMITP with a shimmer current level of 80% at 20 kW. Feedstock was Ti powder with a mean diameter of 27 µm and Al powder with a mean diameter 3 µm. As seen in this figure, almost spherical nanopowder can be synthesized. The mean diameter of the synthesized nanopowder was estimated as 65 nm. The production rate was calculated to be 400 g/h. We also obtained Fe-doped TiO₂ nanopowder with a mean diameter of 61 nm with a production rate of 500 g/h when the feedstock of Fe and Ti powder mixture was used. These obtained production rates are much higher than those by the conventional induction thermal plasma method.

Figure 3 indicates the total diffusive reflectivity %R of the synthesized Al-doped TiO₂, Fe-doped TiO₂ nanopowder and the commercially obtained TiO₂ nanopowder (P-25). The synthesized Al-doped TiO₂ nanopowder has higher absorption than P-25 for UVA region. The synthesized Fe-doped TiO₂ powder has an absorption edge at longer wavelength than the others. It also has a shoulder in %R around 2.5 eV, which is due to Fe doping.

4. Summary

In this report, we have introduced the developed pulse-modulated induction thermal plasma with time-controlled feedstock feeding (PMITP-TCFF) method for a large amount synthesis of nanopowder. Use of this method provides a high production rate around 400 g/h @20 kW for metallic-ion doped TiO₂ nanopowder.

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Fig. 1 The concept of PMITP-TCFF method.



Fig. 2 FE-SEM images of synthesized Al-doped $\mathrm{TIO}_{\mathrm{2}}$ nanopowder.



Fig.3 Total diffusive reflectivity of synthesized nanopowder.