

5th Asia-Pacific Conference on Plasma Physics, 26 Sept-1Oct, 2021, Remote e-conference

## High-rate synthesis of Fe<sup>3+</sup>-doped TiO<sub>2</sub> nanoparticles

using two-coil tandem-type modulated induction thermal plasmas

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Nanoparticles are particles with sizes less than 100 nm, which have different unique properties from bulk materials. For various applications of such nanoparticles, it is desired to develop a high-rate synthesis method for nanoparticles. The authors have developed "a pulse modulated induction thermal plasma (PMITP) with time-controlled feedstock feeding (TCFF)" method, i.e.PMITP+TCFF method, which is one for synthesizing large amounts of nanoparticles [1]. We further developed a tandem-coil MITP, which is sustained by two-coils connected with two different power supplies. This tandem-MITP is expected to have robustness against disturbance from the heavy load feedstock feeding and a large modulation of the coil current. In this study, we investigate the effect of tandem MITP on Fe<sup>3+</sup>-doped TiO<sub>2</sub> nanoparticle synthesis against the single coil MITP.

Experimental conditions were set as follows: The pressure in the chamber was controlled to be 300 Torr. The Ar/ O<sub>2</sub> gas mixture was used as a sheath gas with a 90/ 10 L/min flow rate. The gas flow rate of Ar carrier gas with feedstock and O2 quenching gas was 4 L/min and 50 L/min. The feedstock was a powder mixture of 5 wt%Fe and 95 wt%Ti, whose diameter was less than 45 µm. The modulation cycle of coil current and feedstock feedings were set to both 15 ms. The feedstock powder was supplied intermittently to the plasma torch with feedstock feed rate of 4-8 g/min for single/ tandem-MITP. For single MITP, the time averaged input power was fixed at 25 kW. The coil current was modulated with an 80% duty factor (DF) with on/ off-time of 12/3 ms with modulation ratio of 80%. The longer off-time than 3 ms and lower shimmer current level (SCL) than 80% could not be adopted for stable operation of Ar/ O<sub>2</sub> single-MITP. For tandem-MITP, the time-averaged input power of the upper coil and lower coil were fixed at 15 kW and 8 kW, respectively. The upper coil current was not modulated, while the lower coil current was modulated with on/off-time of 10/ 5 ms. The SCL for the lower coil current was set at 10%.

Fig. 1 shows the mean diameter of fabricated particles ascertained from SEM analysis, and the BET equivalent particle diameter. The tandem-MITP can suppress the particle size more than the single-MITP. This is because the tandem-MITP enables a larger modulation of the thermal plasma than the single-MITP, so that the evaporated vapor can be rapidly cooled. This results in strong supersaturation, nucleation and suppression of condensation and coagulation.

Fig. 2 shows total diffuse reflectivity spectra of the fabricated particles for each condition. The P-25 and doped  $TiO_2$  NPs synthesized by single-MITP and tandem-MITP have high absorption in the ultraviolet (UV) light region because pure  $TiO_2$  has an energy band

gap ( $E_g$ ) of 3.2 eV. In the visible light region, there was almost no light absorption for P-25, whereas reflectivity was lower for doped TiO<sub>2</sub> synthesized by single-MITP. Doped TiO<sub>2</sub> synthesized by tandem-MITP has a further lower reflectivity. In reflectivity curves of TiO<sub>2</sub> synthesized using MITPs, a flat part can be seen around photon energy of 2.5 eV. This flat part appears because of the presence of the sub-band by Fe<sup>3+</sup> doping in TiO<sub>2</sub>. Moreover, doping Fe<sup>3+</sup> to TiO<sub>2</sub> shifts the absorption edge to the visible light range. The results mentioned above show that the TiO<sub>2</sub> NPs synthesized by MITPs can be Fe<sup>3+</sup>-doped TiO<sub>2</sub> NPs, which are anticipated as a visible light responsive photocatalyst.



[1] Y. Tanaka, T. Tsuke, W. Guo, Y. Uesugi, T. Ishijima, S.Watanabe, K.Nakamura, J. Phys. Conf. Ser., 406, 012001, (2012)



Figure 1 Mean diameter of fabricated particles ascertained from SEM analysis and BET equivalent particle diameter.



Figure 2 Total diffuse reflectivity spectra for  $Fe^{3+}$ -doped TiO<sub>2</sub> synthesized for the present study and commercial pure-TiO<sub>2</sub>(P-25).