Fabrication and Evaluation of Composite Photocatalytic Film by Mechanical Coating Technique

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In recent years, investigations on the fabrication and functional characteristics of TiO2 film have been widely reported due to its potential applications as a photocatalyst, gas sensor, solar cell and others. Numerous techniques have been used in the formation/fabrication of films on substrates of various materials with the foremost of these physical vapor deposition (PVD) and chemical vapor deposition (CVD). However, a large and complicated equipment is required, also several of these techniques only operate in high vacuum. In the present study, TiO2 composite film on alumina balls was fabricated by mechanical coating technique (MCT).

First, Ti powders with 99.9% purity and an average diameter of 35 μm were used as the coating metal. Alumina balls with an average diameter of 1 mm were used as substrates. A planetary ball mill (P5/4, Fritsch) was used for MCT. Ti powders of 40 g and alumina balls of 60 g were placed in a pot of 250 ml, and MCT was carried out with a rotation speed of 300 rpm for 10 h. The alumina balls with Ti film obtained from this process are denoted by M10-Ti. Then, alumina balls with Ti film (M10-Ti) of 15 g and TiO2 powders (ST-01, Ishihara Sangyo Kaisha, Ltd., Japan) of 13 g with anatase form and an average diameter of 7 nm were placed in the pot, then MCT was carried out with a rotation speed of 300 rpm for 1, 3, 6 and 10 h. The samples from the above processes are denoted by CM1, CM3, CM6 and CM10, respectively.

The composite film fabricated by the above processes was examined by scanning electron microscope (SEM)(JEOL, JSM-6100) and the crystal form was analyzed by X-ray diffraction (XRD)(JEOL, JDX-3530) spreading the balls with the composite film on a holder.

Photocatalytic activity of the samples with the composite film was evaluated by measuring the decomposition rate of methylene blue (MB) solution (water solution) at room temperature. The samples were spread uniformly on the bottom of a cylinder-shaped cell with φ 20 × 50 mm and 7 ml of MB solution with 10 μM (where μM = μmol l⁻¹) was poured into the cell. photocatalytic activity was evaluated under an intensity of the ultraviolet radiation of 1 mW·cm⁻² for 24 h at room temperature. These conditions were referenced to Japanese industrial standard (JIS R 1703-2). The absorbance of MB solution was measured by a colorimeter (mini photo 10, Sanshin Kogyo) with UV radiation of a wavelength of 660 nm. The gradient, k [nmol l⁻¹ h⁻¹] of the time-MB solution concentration line was calculated out by the least-squares method removing the data of the first 1 h, and is used as the degradation constant.

Figure 1 shows SEM microstructures of the samples. From Fig.1(b)-(c), surfaces of the composite film were formed pores at the convex regions. Especially, many pores were formed at the surface of CM10. From Fig. 1(e)-(f), dark areas, which are TiO2, were formed on concave regions.

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Figure 1 shows SEM micrographs of samples with the composite film. (a), (b) and (c): secondary electron images (d), (e) and (f): back scattered electron images

Figure 2 shows XRD patterns of the samples with the composite film. The peaks of anatase form appeared on 26 deg. and 48 deg. (see Fig. 2(c)-(f)). The intensity of anatase TiO$_2$ increased with increasing of milling time, but decreased with increasing of milling time in the case of over 3 h. It hints that the amount of TiO$_2$ of the composite film reached maximum at 3 h, then the amount of TiO$_2$ decreased because of the effect of the abrasive wear. From the above results, it is found that the composite film having a composite microstructure of TiO$_2$/Ti/Al$_2$O$_3$ was formed through the above processes.

The degradation constant, $k$ shows in Fig. 3. It is found that the degradation constant goes up and reaches a peak around milling time of 3 h (CM3), and then decreases with increasing milling time.

In the present study, MCT is simple and economic to fabricate the composite film of TiO$_2$ and Ti. The composite film on alumina balls has a microstructure of TiO$_2$/Ti/Al$_2$O$_3$, and TiO$_2$ has anatase form. Photocatalytic activity goes up with milling time and reaches a peak around milling time of 3 h, and then goes down.

Figure 3 Relationship between milling time and degradation constant.
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