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Feature article

AZO photocatalytic coating deposited by plasma thermal spraying with shell-type feedstock powder

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ABSTRACT

Aluminum zinc oxide (AZO) was deposited on a glass substrate using plasma spraying technique. Hierarchical nanostructured morphology is observed, for the first time, in plasma spraying coatings. Nanorod and nanosphere adhere to micro spherical particle to form hierarchical structure, however, is only reported by chemical synthesis method. Through XRD and SEM analysis, it is found that the morphology of coating can improve the formation of defects greatly. After UV-A light testing, photocatalytic efficiency of coating was analyzed using UV-vis spectrometer. Combined with photoluminescence and X-ray photoelectron spectroscopy analysis, the results indicates that AZO coating is with good enhanced photocatalytic properties. This phenomenon suggests that AZO crystallites consist of multiple facets with high surface-energy planes. The measured water contact angle of about 7° indicates that AZO coating is with high hydrophilic characteristic and has very good practical applications in industry. This study thus paves a new way for environmental pollution control.

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1. Introduction

In recent years, reducing environmental pollutant is currently in great demand, and this issue of research mostly focuses on the use of photocatalysts. Owing to significant advantages including high degradation ability for organic compound under ultraviolet (UV) light or sunlight and low cost, photocatalysts in contaminated air or water treatment work have been extensively studied for the last 40 years [1].

ZnO has been gradually replacing commercial TiO_2 as a photocatalyst because of its similar energy gap (E_g is \sim 3.3 eV at 300 K) [2–5], lower cost, and easy accessibility for research and development. Additionally, ZnO adopts a unique phase under a normal atmosphere [6]; however, under the same atmosphere, phase transitions of TiO_2 , with three phases, can occur at different temperatures, but only the major anatase phase of TiO_2 is useful as a photocatalyst [7–9]. Thus, the relative preparation superiority of ZnO can enable the use of higher synthesis and subsequent heat treatment temperatures (e.g., >500 $^{\circ}$ C). Aluminum- or hydrogendoping of pure ZnO materials has been successfully used to form n-type ZnO (donors), and these elements exhibit a significant effect

Control of ZnO powder morphology such as hierarchical structure and exposure of facets by using chemical method is reported and would help for enhancing their catalytic activity and selectivity [9,16,17]. Although photocatalytic powders provide great efficiency due to their higher specific surface area compared to their corresponding films, they are limited in ordinary and industrial applications. However, the hierarchical structure of aluminum zinc oxide (AZO) film is still rarely reported.

This research focuses on the fabrication of AZO with a high photodegradation contact surface and a nanocrystal structure via plasma spraying. A related analysis of organic compound degradation or removal property is also addressed. The as-sprayed AZO

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on majority carrier concentrations [10]. Moreover, doping elements act to enhance and stabilize the electron flow from intrinsic defects in n-type ZnO, such as oxygen vacancies and Zn interstitials. Photocatalytic efficiencies through UVA (ultraviolet A) or white light beams are also dependent upon specific surface areas [11,12]. Nejand et al. used ZnO as a barrier layer between a TiO₂ photocatalyst and soda-lime glass due to sodium diffusion into the TiO₂ film and interferences with the formation of the anatase phase [13]. The Na ions being as inhibitors of the photocatalytic activity of the TiO₂ films also reported by Tomaszewski et al. [14]. Li and Haneda also found that the microstructure, crystal morphology, and specific surface areas of ZnO directly controlled the improvement in photocatalytic efficiency [15].

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photocatalytic coatings consisting of nano- and microparticles, particularly for the relationship among deposition morphology, crystallization, and film formation mechanism are studied. Also the coating optical properties including its defects and bandgap are investigated.

2. Materials and experimental methods

Aluminum zinc oxide powder with particle size of 5-45 µm (Yong-Zhen Technomaterial) was used in this study. The ideal composition of the powder was 97 wt.% ZnO and 3 wt.% Al₂O₃. The specific surface area (BET) of the powder was 50-70 m²/g. Thermal spraying was used to deposit proper thick coatings on soda-lime glass substrates for the fabrication of metallographic specimens. An F4 plasma spraying gun (Sulzer Metco, Winterthur, Switzerland), using 41 SLPM argon and 11 SLPM hydrogen as the plasma gases, was used to produce the thermal spray coatings. The spraying parameters are shown in Table 1. According to the robot speed and the powder feed rate, the layer thickness deposited per pass was about 3 µm in this work. The total number of passes was set to 20. All samples were then ground and polished before analyzing the microstructure through metallurgical microscopy. An epoxy resin that had been treated with glass sand, which helps to increase the hardness of the resin, was used for sample preparation. A JEOL JSM-6500F FESEM (field-emission scanning electron microscope) was used to analyze the microstructure of the thermal spray coatings.

X-ray diffraction (XRD), scanning electron microscopy and energy-dispersive spectrometry (EDS) were also used to analyze the microstructures of the thermal spray coatings. For the XRD measurements, a Phillips PW3710 vertical goniometer was used to characterize the structures of coatings that had been sprayed, but a Phillips PW1710 X-ray diffractometer was used to characterize the structures of powders. The texture coefficient of powders and coatings was measured by corresponding XRD soft (MDI Jade 5.0). The diffraction patterns of AZO powder and coating samples were also simulated by Maud software, version 2.33. This software is based on the Rietveld full pattern refinement method [18].

According to carefully refinement analysis from each separate XRD patterns of AZO powder and coating samples, superimposing the simulated profiles with corresponding to the experimented profiles achieved almost the indistinguishable differences. And their weighted residual error (Rwp) and goodness values are given by less than 15% and approximate 1, respectively. In simulated operation, the triaxial stress isotropic model [19] was chosen to fit lattice strains for samples. For calculation of triaxial stress of AZO, the related Young modulus's and Poisson's ratio are set as 128 GPa and 0.35 at temperature 300 K, respectively, which are from the ZnO bulk material [20]. The directions of σ_{11} , σ_{22} , and σ_{33} stresses are parallel to the directions of X_1 , X_2 , and X_3 axes respectively in the orthogonal coordinate system [21]. And these stress directions correspond with the directions of a_1 , a_2 , and c axes in hexagonal closest packed (HCP) unit cell, respectively.

X-ray photoelectron spectroscopy (XPS) was utilized to further analyze the chemical composition of the AZO coatings. XPS was conducted using a Thermo VG ESCAlab 250 (ThermoVG Co.) with an Al (K_{α}) X-ray source. The X-ray photoelectron spectra of the samples were calibrated using the carbon 1 s peak (284.6 eV). The surface of the APS AZO coating was cleaned using Ar $^{+}$ ion sputtering for 300 s, whereas that of the AIP-Ref AZO coating was for 120 s. The two sample codes (APS AZO coating and AIP-Ref AZO coating) are described below (see Table 2).

An arithmetical mean roughness (Ra) value of the coating was used as the index, which can be measured using a Mitutoyo Surftest-401 surface roughness analyzer.

This study also focused on the application of UV light as an alternate source for the photocatalytic degradation of methylene blue (MB), the agent commonly used as a standard organic compound for photocatalytic reactions [22,23]. A flexible desktop darkroom fashioned as a square-shaped iron case was used to perform the photocatalytic degradation of methylene blue dye. The inside of the iron case included a UV source (Philips TL8W/08 × 4, UVA 300–400 nm (peak on 360–370 nm)), cooling fans and an AZO coating $(2.5 \times 2.5 \text{ cm}^2)$ immersed within 16 ml of methylene blue dve solution (initial concentration: 1×10^{-5} M) in a glass dish located on the sample stage. The UV source was arranged on the interior roof of the iron case and over the glass dish. A fixed intensity $(1000 \pm 100 \,\mu\text{W/cm}^2)$ radiated through the quartz glass coverplate onto the MB solution in the glass dish. The UV intensity conditions simulated sunlight from a window, and the conditions were similar to 1 h under outdoor UV light intensity, based on previous research [24]. The photocatalytic efficiency ($\frac{(C_0-C_t)}{C_0} \times 100\%$, C_0 is the initial normalized concentration of MB and C_t is the normalized concentration of MB after the period of radiation time) of the AZO coatings was evaluated using the UV radiation in this darkroom, i.e., the degradation of the MB dye after proper radiation time was confirmed using a UV/VIS spectrophotometer (HATACHI U-3010). The absorption (Abs) value refers to the maximum peak of 665 nm in the UV/VIS spectrophotometer. This wavelength was primarily absorbed by the MB. Note that the UV illumination test used clean MB solution for each sample every time.

And, the diffuse reflectance measurement was performed using UV-vis diffuse reflectance spectroscopy (UV-vis DRS) with integrating sphere (HATACHI U-3010). According to the Kubelka-Munk equation [25,26], absorption coefficient α can be given by:

$$F(R) = \frac{K}{S} = \frac{(1-R)^2}{R} = \alpha \tag{1}$$

$$[F(R) \cdot E]^{m} = (\alpha h \nu)^{m} = C_{1}(h\nu - E_{g})$$
(2)

where K is the K-M absorption coefficient, S is the K-M scattering coefficient, R is the diffusion reflectance data as a function of wavelength (λ), E (equal to h ν) is the photon energy, C₁ is the proportionality constant, α is the linear absorption coefficient of the material, and E_g is the band gap of a powder/coating sample. Thus, the energy gaps E_g were calculated by the function of [F(R)·E]^m against energy E. Here, the value of the exponent m equals 2 for AZO material, whereas m equals 1/2 for TiO₂ material. Photoluminescence (PL) measurement was performed using the 325 nm line from a Xenon pulse lamp as the excitation source (HATACHI F-4500 FL Spectrophotometer).

Surface wettability was analyzed with static contact angle measurements against water (H_2O) using a FACE Measurement and Analysis System (FAMAS). Before the contact angle (CA) test, all the sample surfaces were cleaned with deionized (DI) water and blown dry with compressed air and then hot air. H_2O droplets of 47.5 mm in diameter were used each time, and the average value of the contact angles was obtained at ten different locations on the sample surface. A significant hydrophilic characteristic is indicated by contact angles less than 10° [24].

At present study, different powders and coating samples including four test samples and three reference samples were used in the experiment (Table 2). Those coatings including (1) AIP (Arc ion plating)-Ref AZO (**Fig. A.1**), (2) TiO₂ spray coating by hand-held pressurized sprayer, and (3) APS (Air plasma spraying) AZO coating were used. The phase composition of TiO₂ P25 powder contains about 80 wt.% anatase and 20 wt.% rutile typically. Its particle size and BET were 21 nm and $50 \pm 15 \, \text{m}^2/\text{g}$, respectively. The particle size of TiO₂ sprayer coating on a glass substrate was 30–110 nm and its coating thickness was near 440 nm (**Fig. 1**). The AIP-Ref AZO coating was also deposited on a glass substrate and its coating thick

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